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TEXTILE RESEARCH JOURNAL

VOL. XXVI, NO. 12 DECEMBER 1956

Static Electrification of Filaments

Theoretical Aspects[1]

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Abstract

A theoretical model based on the band structure of the solid state is proposed in order to describe the transfer of charge when two solids are rubbed together. Only the structure in the interior is considered, the interpenetration of the bodies being supposed deep enough that the effects of foreign surface layers and of surface states are slight. The theory is applied to data on static electrification of filaments rubbed together under controlled ambient and mechanical conditions. The theory provides a semiquantitative description of most of the observed phenomena.

I. Introduction

An earlier paper [7] described the experimental techniques and the results of a study on the static electrification of filaments rubbed against one another under controlled mechanical and ambient conditions. The present paper proposes a semiquantitative theory of the mechanism of charge transfer under these conditions, and discusses the experimental results in the light of this theory.

It is too much to hope for a complete elucidation of the phenomena of static electrification at the present state of knowledge. On the experimental side, the microscopic geometry of the contacting surface is not known, the chemical nature of the surfaces is not certain, and transient temperatures and pressures at the region of contact are unknown. On the theoretical side, it is not clear that sufficient periodicity occurs in the usual materials of interest to allow application of much of the modern theory of solids, and it certainly appears that very accurate quantitative treatments cannot be made because of the prohibitive labor of the calculations and the unavailability of data on energy levels. Nonetheless, we believe that some progress can be made, and we shall set as our goal a description of the phenomena on a simplified picture, namely, the transfer of electrons between two contacting, almost perfectly periodic lattices, and the retention of part of the transferred charge upon separation.

[1] This paper is based on a part of the dissertation submitted by S. P. Hersh in partial fulfillment of the requirements for the degree of Doctor of Philosophy at Princeton University.

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In Section II the assumptions for the theoretical treatment are detailed, and their application to the problem at hand is made. In Section III the experimental results are discussed in terms of the theory. In Section IV our conclusions as to the likely worth of this picture are stated.

II. Assumptions and Analysis

A. It is assumed that *electrons only are transferred.* Although our experiments give no direct evidence on this point, we believe that the high mobility and the relative ease of detachment of electrons, compared with ions, make it gratuitous to postulate significant ion transfer. In this we agree with Frenkel [3] and with Harper [5]. The latter states "Electrolytic potentials . . . although they can no longer be thought of as an *alternative* to intrinsic contact potentials . . . may easily be present as a *complication* in some circumstances."

B. It is assumed that *the direction of charge transfer is determined by the relative position of the Fermi levels in the two contacting materials, the transfer taking place so as to tend to equalize the levels.* In equilibrium, the levels must be equal, as can be shown by statistical mechanics; before equilibrium, kinetic theory indicates that electron flow occurs so as to tend to make the levels equal.

C. It is assumed that *the amount of charge transferred during contact is determined by the energy band structure of the contacting materials.* This question must be examined at some length. We assume first that forbidden and allowed energy bands do exist; on general quantum-mechanical grounds it would be surprising if they did not. But we assume further that enough periodicity or near-periodicity exists so that the usual concepts and language of the band picture can be applied. We assume next that in most rubbing contacts of any severity the charge transfer is controlled by the properties of the interior of the crystal, rather than by the properties of the surface. In particular, we believe that absorbed gases and other contaminants have little effect (unless of course a hard oxide layer is formed), because in most contacts the forces are so large that the regions of interatomic interaction are primarily beyond the original surface layers of the contacting objects. One consequence of this assumption is that surface states,[4] i.e., states at a crystal-vacuum interface, are of slight importance so far as the process of initial charge transfer is concerned. However, a new assumption is entailed, namely, that "interface states," i.e., possible states existing at crystal-crystal interface, are of minor importance in controlling the charge transfer. For brevity, we limit ourselves to metals and insulators, considering intrinsic semiconductors simply as a special case of the latter, and believing that impurity semiconductors can be included by natural extension of the picture.

Substances in Isolation

According to modern theory of the solid state, the periodic structure of the potential field results in a band structure for the energy zones, with the electrons having quasi-continuous sets of allowed energies separated by regions of forbidden energies. These allowed bands are then populated according to Fermi-Dirac statistical laws. Figure 1*a* represents the arrangement of energy bands in an isolated metal, and Figure 1*b*, that in an isolated insulator.

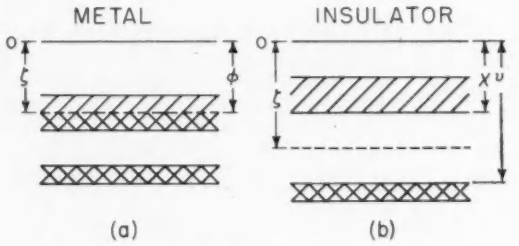

Fig. 1. Schematic representation of energy levels in metal and in insulator.

Here the potential energy of an electron is plotted against distance within the crystal. The zero of energy is referred to a stationary electron at an infinite distance from a neutral crystal. Ordinarily any distance greater than ten or a hundred Angstrom units may be taken as infinite, and we shall use the expression "outside the crystal" to indicate regions farther than this. In Figure 1 are shown

[4] By "surface states" we refer explicitly to those additional states arising from the disturbance of periodicity at the end of one crystal. There may be other states, localized at the surface or within the body of the crystal, due to other imperfections of one sort or another; in fact, there must be to provide immobilization of charge. But these "imperfection states" need—and in fact must, on the basis of our picture—lie only a small amount below the usual "volume states" given in the band picture. It is true, of course, that our picture would not be changed in its essentials even if the flow of charge were into surface states. But we have even less basis for prediction of these levels.

the uppermost bands in accordance with the usual picture. In the insulator are shown the uppermost nearly empty band and the uppermost nearly full band. In the metal are shown the uppermost half-filled band and the uppermost nearly filled band. For the insulator the Fermi level lies at an energy ζ almost midway between the nearly full and nearly empty bands, and for the metal it lies almost midway in the half-filled band.

In the insulator we shall be concerned with the energy χ released when an electron at rest outside the crystal is taken into the lowest level in the uppermost nearly empty band. This quantity is the analog of the electron affinity A for a free molecule.[5] We shall be concerned also with the energy v necessary to remove an electron from the highest level of the uppermost nearly filled band. This quantity is the analog of the ionization potential I for a free molecule.[5] In the metal we shall be concerned with the energy ϕ released when an electron at rest outside the crystal is taken to the Fermi level, or absorbed when an electron is taken from the Fermi level to a point outside the crystal. In a sense ϕ may be considered the analog of either the electron affinity or the ionization potential for a free molecule. It has received the special name, "work function."[6]

Substances in Contact

When two substances which in isolation have different Fermi energies are placed in contact, electrons will flow from the one with higher ζ to the one with lower ζ, so as to tend to equalize the Fermi levels. In the case of metal–metal contacts, the relaxation time for this transfer is extremely short, and the transfer may be considered instantaneous. In the case where one (or both) of the contacting materials is an insulator, the relaxation time may be very long at room temperature, and for good insulators it is unlikely that equilibrium is approached closely in periods shorter than years.

[5] The quantity χ is frequently called "electron affinity" in the literature of the solid state, but we should prefer some designation differentiating it from the quantity A for a free molecule. The quantity v does not seem to have been called "ionization potential," though the reasons for such a designation are just as cogent as those for "electron affinity." We wish neither to continue what is to us an undesirable terminology, nor to introduce a new one at this time, and shall simply employ the appropriate symbols.

[6] For a more precise definition of work function and a discussion of other definitions of this quantity, see Herring and Nichols [6].

The qualitatively different cases of contact between metals and insulators are sketched in Figures 2, 3, and 4 for the case of two semi-infinite materials making contact at a plane surface. The diagram to the left in each figure represents the energy bands before contact. The diagram to the right shows the energy bands at equilibrium after contact. Here all the energies are referred to a zero taken as the potential energy outside the left-hand body at a large distance from the contact surface. Enough charge is transferred to bring the Fermi level in the right-hand body to the Fermi level in the left-hand body. There is the additional condition that at large distances from the contact surface the energy bands must be the same as for the isolated bodies. The detailed dependence of the potential with distance is calculated by setting up Poisson's equation with the charge density given by the application of Fermi-Dirac statistics to the electrons occupying the allowed energy bands (see, for example, Mott and Gurney [11], pp. 170–171; Fan [2]).

Metal–Metal Contact. Figure 2 represents the contact between metals. Electrons in the half-filled band in the left-hand metal spill over into the half-filled band in the right-hand metal. There are many electrons available, and many vacant levels for them to go to. Hence large charge[7] (of the order of 10^4 esu/cm.^{2}) is lost by the left-hand metal from a small spatial region (of the order of one Angstrom unit thick), and an equal charge is gained by the right-hand metal within a small spatial region.

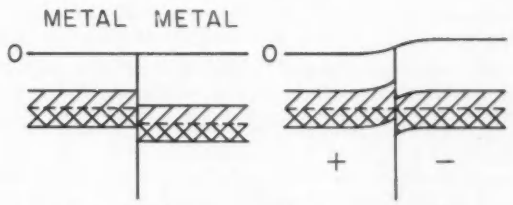

Fig. 2. Schematic representation of energy levels near metal–metal interface, before and after contact.

Insulator–Insulator Contact. Figure 3*a* represents the contact between insulators when in isolation a filled band in one material does not overlap an empty band in the other. Electrons can escape from the left-hand insulator to the right-hand one only by thermal agitation from a filled band into an empty band at a higher energy. Hence a small charge (perhaps 10^{-1} esu/cm.^{2}) is lost by the left-hand insulator from a large spatial region (perhaps 10^6 A thick), and an equal charge is gained by the right-hand insulator within a large spatial region.

Figure 3*b* represents the contact between insulators when in isolation a filled band in one material overlaps an empty band in the other. Electrons can now spill from the filled band of the left-hand material into the empty band of the right-hand material. Hence a large charge (of the order of 10^4 esu/cm.^{2}) is lost by the left-hand insulator from a small spatial region (of the order of 10 A thick), and an equal and opposite charge is gained by the right-hand insulator within a small spatial region.

[7] This estimate and subsequent ones are on the basis of calculations by Fan [2], Mott and Gurney [11], or Van Ostenburg and Montgomery [13].

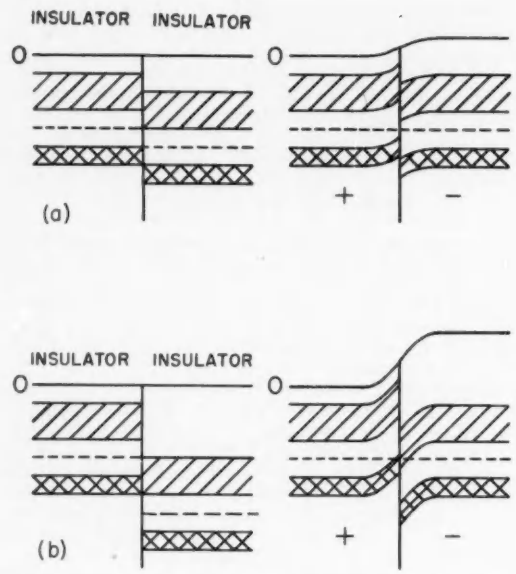

Fig. 3. Schematic representation of energy levels near insulator–insulator interface, before and after contact.

Metal–Insulator Contact. Figure 4*a* represents the contact between metal and insulator, when in isolation the Fermi level in the metal lies below the Fermi level in the insulator, and when the top of a filled band of the insulator lies above the Fermi level in the metal. Then electrons can spill from the filled band in the insulator to the half-filled band in the metal. Hence much charge is lost by the insulator from a small spatial region, and much gained by the metal within a small spatial region. Accordingly the potential changes in each material are of comparable magnitude.

Figure 4*b* represents the contact between a metal and an insulator, when in isolation the Fermi level in the metal lies below the Fermi level in the insulator, and when the top of the filled band of the insulator lies below the Fermi level in the metal. Then electrons can escape from the insulator only by thermal agitation from the filled band in the insulator to the half-filled band in the metal. Hence a small charge is lost by the insulator from a large spatial region, and an equal charge gained by the metal within a small spatial region. Accordingly most of the potential change occurs within the insulator.

Figure 4*c* represents the contact between a metal and an insulator, when in isolation the Fermi level in the metal lies above the Fermi level in the insulator, and when the bottom of the empty band of the insulator lies above the Fermi level in the metal. Then electrons can escape from the metal only by thermal agitation from the half-filled band in the metal to the empty band in the insulator. Hence a small charge is lost by the metal from a small spatial region, and an equal charge is gained by the insulator within a large spatial region. Accordingly most of the potential change occurs within the insulator.

Figure 4*d* represents the contact between a metal and an insulator, when in isolation the Fermi level in the metal lies above the Fermi level in the insulator, and when the

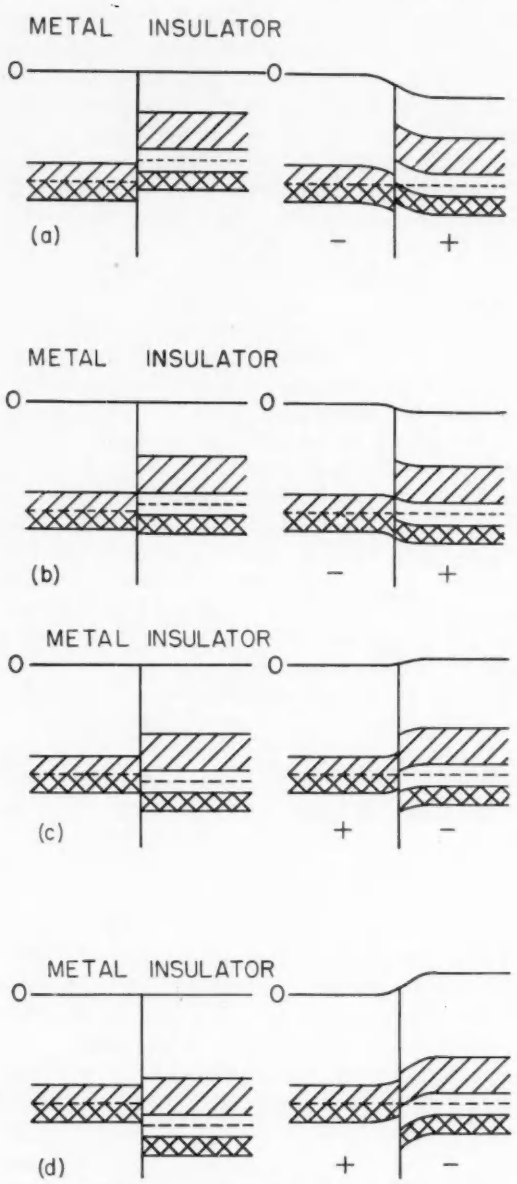

Fig. 4. Schematic representation of energy levels near metal-insulator interface, before and after contact.

Fermi level in the metal lies above the bottom of an empty band in the insulator. Then electrons can spill from the half-filled band in the metal into the empty band in the insulator. Hence a large charge is lost by the metal from a small spatial region, and much gained by the insulator within a small spatial region. Accordingly the potential changes in each material are of comparable magnitude.

The discussion just given applies to equilibrium. In the case of metal–metal contacts, equilibrium should be attained instantaneously, with transfer of large amounts of charge per unit area of metals in contact. In the cases where one of the substances is an insulator, and electrons can spill freely from occupied levels to empty levels, the charge transfer near the interface should take place nearly instantaneously. However, as can be seen from the diagrams (Figures 3*b*, 4*a*, 4*d*), an additional charge is furnished by thermal agitation to the empty band away from the interface. While this charge is not large, it is appreciable. Hence a small portion of the transfer may take significant time. In cases where one of the substances is an insulator, and electrons cannot spill freely from occupied levels to empty levels, we should be prepared to have to wait a long time for equilibrium, with transfer of only small amounts of charge. Only if conductivity is appreciable, if the energy differences are very small, or if temperature is very high, will we expect much charge transfer in the cases shown in Figures 3*a*, 4*b*, and 4*c*.

D. It is assumed that *the area of "true contact," that is, the area of interpenetration of atomic fields, depends only on the normal force between the objects rubbed.* This assumption finds its support in the conclusions of Bowden [1] on the nature of frictional contacts. It is true that Bowden states this conclusion for metals only, but we believe that the main features will apply to nonmetals also, in particular those devoid of a highly crystalline structure. For relatively high loads we believe that increase in the true contact area is proportional to the increase in load, but for very light loads we are not sure that this law would be followed. The reason for this doubt is that, during sliding, the interpenetration of atomic fields on the slant surfaces of the asperities—which presumably determines the amount of charge transferred—may have a different dependence on the normal force from that of the deformation of the asperities. Actually we have made no experiments at very light contacts, and we can neither support nor refute speculation as to what happens under these conditions.

E. It is assumed that *after separation the transferred charge is localized at the place of contact in insulators, but not of course in metals.* The first part of this assumption is to some extent *ad hoc*. On the experimental side there is no doubt that charge can be localized in insulators. But according to the theory of the solid state, electrons inserted in an empty band, or holes left in a filled band, are free to move throughout the crystal, once the attractive forces resulting from the charge of opposite sign on the other material are removed. Gonsalves [4] has used this argument to formulate a theory based on surface states. This is certainly a possibility, but we believe that the postulation of surface states is unnecessary, since the existence of surface states provides for a localization of electrons only in certain cases; in some cases where it appears from the theory that electrons should be free to move on the surface, they are found to be fixed. Some mechanism of localization must be postulated, and this is usually that of imperfections on the surface (*cf.* discussion in Seitz [12], pp. 325–326). Now several mechanisms to trap electrons at the surface are recognized (*cf.* discussion in Mott and Gurney [11], pp. 86–88, 124–131), and though we are not in a position to suggest any definite mechanism, we believe that by some means charge can be immobilized within the volume of insulators.

Actually we shall be dealing with neither perfect conductors nor perfect insulators. Metals can be considered as perfect conductors so far as electron mobility is concerned, and the better insulators—amber, polystyrene, polyethylene, Teflon—can be considered as perfect insulators. But with many materials of intermediate conductivity—nylon, cotton, wool—the classification depends on the time scale of the experiment. The charge distribution will decay after separation according to the laws of electrodynamics, and one may expect to find that during rubbing, the charge can move over regions larger than the area of contact, but still not all the way along or around the filament.

F. It is assumed that *upon separation of the surfaces after contact the charge initially transferred is reduced by tunneling through the gap formed by the separating surfaces.* Harper [5] has made an excellent study of this process for spheres of metals possessing different work functions. He finds that the fraction of charge remaining is nearly independent of speed of separation, and from his analysis it may be concluded that this fraction is of the order of one-half. He does not analyze the insulator-conductor case, but it seems clear that the fraction will be reduced from the conductor–conductor case, because the inability of the charge remaining on the insulator to redistribute itself will keep the driving field up during the separation. But we suspect that the fraction will not decrease below one-tenth, even for insulator–insulator contacts. This point deserves further investigation. Our geometry (crossed cylinders) differs from Harper's (spheres), but we do not see that this effect will be important.

G. It is assumed that *under an important set of circumstances the charge remaining after separation is not neutralized significantly by ions resulting from atmospheric breakdown.* This is really a restriction on the range of the applicability of the theory, not an assumption. Specifically, we believe that for the experiments described in reference [7], except possibly for those at the highest normal forces, the effect of breakdown is negligible. A discussion of our reasons for this belief has been given in reference [7], pp. 290–291, which we may here spare the reader.[8]

III. Discussion of Results

A. *Direction of Electron Transfer*

According to the considerations outlined earlier, the direction of charge transfer depends only on the relative positions of the Fermi level. Hence a unique triboelectric series should exist for all materials. This is a valid deduction, if the definition of "material" is made specific enough. First of all the body must be sufficiently homogeneous for it to be considered as constituting one material. We believe that for contacts severe enough to cause surface penetration, many more materials satisfy this criterion than had been thought in the past. But even then it must be realized that the position of the Fermi level depends not merely on chemical composition, but on temperature and on the detailed molecular structure. Thus we are not surprised that Jamieson [9] noted in 1910 that mechanical strain modifies the charging of celluloid sheets and some other materials, and that in reference [7] an undrawn nylon filament was found always to acquire a negative charge when rubbed against a drawn nylon filament from the same melt. Because of possible inhomogeneities in chemical composition and molecular structure, then, we shall expect consistent direction of transfer from one rub to another, or from one sample to another, only if the composition and structure are such that the Fermi levels in isolation have a reasonably wide separation.

In reference [7] the following triboelectric series for insulators on insulators was found:

POSITIVE
Wool
Nylon
Viscose
Cotton
Silk
Acetate
Lucite
Polyvinyl alcohol
Dacron
Orlon
Polyvinyl chloride
Dynel
Velon
Polyethylene
Teflon
NEGATIVE

The fact that such a series can be established at all lends support to our hypothesis. The series is quite definite, and the evidence is against any cyclical nature of the series.

But one would like to be able to predict the position of a substance in the series, not merely know that a series exists. Estimates of the positions of energy levels, based on either theory or experiment, are scarce even for simple substances (*cf.* Mott and Gurney [11], pp. 75 ff., 245). In the case of certain fibers we have speculated along the following lines. The Fermi energy ζ for an insulator is about half the sum of v and χ. What we should like to do is to estimate v and χ from the ionization potential and the electron affinity, respectively, of the constituent molecules. Our justification for this is that in the direction of orientation of a fiber, that is, along the axis of the fiber, a long chain of atoms is connected by primary valence bonds, with large interpenetration of electron charge densities. Transverse to the axis of the fiber, the cohesive forces consist of secondary valence bonds between atoms of different chains, with only slight interpenetration of electron charge densities. Thus the properties of the constituent atoms of a chain are much affected by being built into a chain (formation of primary valence bonds), but the properties of the chains themselves are but little affected by being built into an aggregate (formation of secondary valence bonds). Consequently there is some hope that the ease of losing or gaining electrons will be controlled largely by the properties of the individual constituent chains, and not by the manner in which they are built into the aggregate. Thus v for a fiber, an aggregate of long chain molecules, on this picture would be ex-

[8] It is probably idle to speculate on this point. The question is being investigated directly by R. G. Cunningham at Michigan State University, who uses a photomultiplier tube placed near the separating surfaces to detect electrical discharge. His preliminary results show that for at least some of the conditions of the studies in reference [7], no appreciable breakdown exists.

pected to be nearly equal to the ionization potential for one of the long chain molecules, and χ for a fiber would nearly equal the electron affinity of one of the molecules.[9]

Unfortunately, there exists little direct knowledge of the ionization potential of long chain polymers, and even less of the electron affinity. We fall back on the assumption that in the absence of severe resonance effects, the electron cloud density around an individual atom is affected seriously only by its very close neighbors. On this basis the electronic properties of a high polymer would differ little from those in a low polymer based on the same monomer, and in some cases they would differ but little from those of the monomer itself. We might expect further that some constituent atom or grouping loses an electron more readily than any other grouping, and thus determines the minimum energy necessary to remove an electron. If this property would be preserved from one substance to another, then a basis for prediction of ionization potential would be available.

As an example, consider the ionization potentials of the series of paraffin hydrocarbons C_nH_{2n+2}. The data of Honig [8] are plotted in Figure 5, where it can be seen that the ionization potential decreases with increasing chain length, and after 7 or 8 carbon atoms levels off to about 10.2 e.v. We take this value then as an approximation to v for polyethylene. By assuming that the ionization potentials of other series, such as vinyl chloride, divinyl chloride, trivinyl chloride . . . polyvinyl chloride, behave in the same way as the hydrocarbon series, we can say that the ionization potentials of the polymers will be ordered in the same manner as the ionization potentials of the saturated monomers. The ionization potentials of the saturated monomers of several materials are listed in Table I, according to the data of Morrison and Nicholson [10]. In parentheses there have been added our estimates for 1, 1-dichlorethane, the principal saturated monomer of Velon, which should have an ionization potential somewhat greater than that of ethyl chloride, since I for chloroform, 11.5 e.v., is greater than I for methyl chloride, 11.2 e.v.; and for perfluorethane, the saturated monomer of Teflon, which should have an ionization potential greater than that of ethane, in view of the high electronegativity of fluorine.

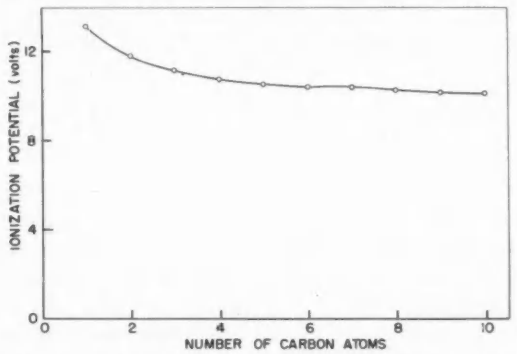

Fig. 5. Ionization potential of the paraffin hydrocarbons C_nH_{2n+2} as a function of the number of carbon atoms.

[9] This behavior is, of course, completely different from that of metals, where building of atoms into a lattice results in high interpenetration of electron densities, with the result that the work function of the solid metal is much different from the ionization potential of the isolated atom.

TABLE I. Ionization Potentials of Saturated Monomers of Some Fibers

Polymer	Monomer	I (e.v.)
Polyvinyl alcohol	Ethanol	10.60
Polyvinyl chloride	Ethyl chloride	11.18
Velon	1,1-dichlorethane	(11.4)
Polyethylene	Ethane	11.76
Orlon	Ethyl cyanide	11.85
Teflon	Perfluoroethane	(12)

There is almost no information available on the electron affinity of the monomers under consideration. Nevertheless, if we assume that $\zeta = \frac{1}{2}(v + \chi)$ decreases in the same order that v does from one monomer to another, then we can expect the triboelectric series to follow the same order as in Table I. Indeed it does, except for Orlon, which is seriously out of place. However, Dynel, a copolymer of acrylonitrile and vinyl chloride, does lie below polyvinyl chloride, and this circumstance suggests that this particular sample of Orlon may contain some ingredient other than ethyl cyanide which loses electrons readily.

With metal–insulator contacts, the hypothesis that direction of charge transfer is controlled by relative position of Fermi levels should be capable of a more stringent test, because only one parameter need be known for a metal, namely ϕ, in place of the two, v and χ, for an insulator. Furthermore, independent data are available on work functions, although in general they cannot be expected to apply directly to the experiments reported. Apart from the matter of surface contamination, it appears that the dipole

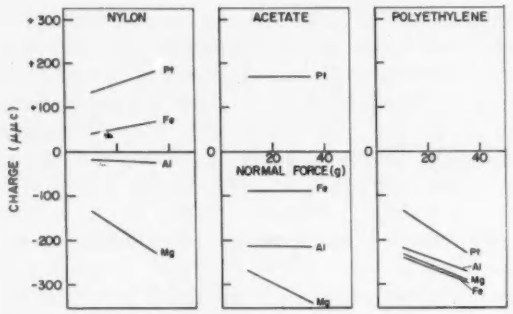

Fig. 6. Charge generated by rubbing scraped metals on insulators as a function of normal force. The charge given is that developed on the insulator.

layer at the metal-vacuum interface in photoelectric or thermionic emission experiments will be different from the dipole layer at the metal-insulator interface in static electrification experiments. Nonetheless, we hope that the work functions determined, say photoelectrically, can be taken as a measure of the Fermi energy. According to [6], the photoelectric work functions in electron volts for platinum, iron, aluminum, and magnesium are about 6.2, 4.6, 4.4, and 3.6, respectively. Figure 6, reproduced from reference [7], shows the results of rubbing freshly scraped wires of these metals on nylon, acetate, and polyethylene filaments. If the Fermi energies of these three insulators are taken as 4.5, 5.1, and 8.2 e.v., respectively, then the sign of charge can be accounted for. These assignments are of course not unique, and in the next section we shall return to this question.

B. *Amount of Charge*

In this section a semiquantitative application of the theory is made. As an introduction to what we have in mind, consider the results on rubbing metals on insulators, as just presented in Figure 6, and of rubbing these insulators on one another, as shown in Figure 7, reproduced also from reference [7]. These results may be pretty well correlated by assigning energy levels as shown in Figure 8. Here the work functions for the metals have been given their photoelectric values, and polyethylene has been assigned the value 10.0 e.v. as a guess based on the value 10.2 e.v. extrapolated from the ionization potential data for the paraffins. When Figure 8 is interpreted in the light of Figures 3 and 4, agreement is obtained in every case for the direction of charge transfer, and in most cases for the magnitude. For example, with the metals on nylon (left-hand graph of Figure 6), the Fermi level for Mg lies above the bottom of the nearly empty band for nylon, and hence a large number of electrons are lost from the metal. The Fermi level for Al lies below the nearly empty band, however, and so only a small charge is lost from the nylon. The Fermi level for Fe lies below that for nylon, but not so low that electrons from the filled band in the nylon can spill into the metal; thus only a small number of electrons are gained by the metal due to thermal excitation. Finally for Pt, electrons can spill freely from the nearly full band of the insulator into the half-filled band of the metal.

The quantitative agreement must not be pushed too far because other factors enter—the conductivity,

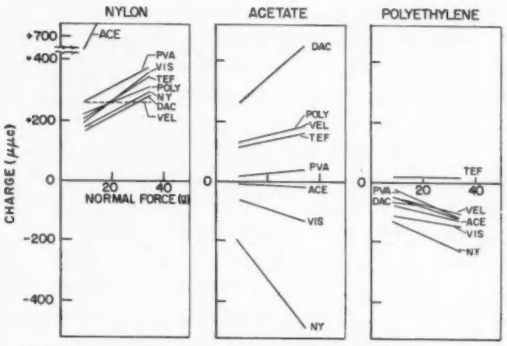

Fig. 7. Charge generated by rubbing various insulators on nylon, acetate, and polyethylene as a function of normal force. The charge given is that developed on the bottom filament. ACE-acetate, DAC-Dacron, NY-nylon, POLY-polyethylene, PVA-polyvinyl alcohol, TEF-Teflon, VEL-Velon, VIS-viscose.

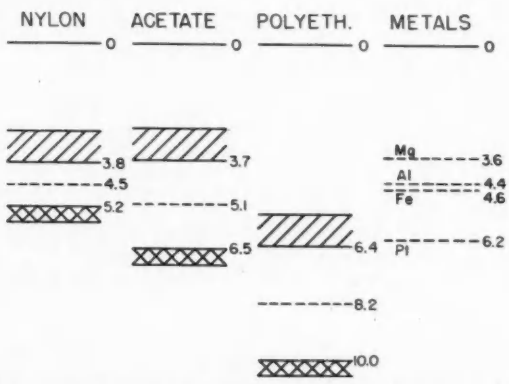

Fig. 8. Diagram of energy levels assumed for materials used in experiments of Figures 6 and 7.

the hardness of the materials, and so on. There is one serious disagreement among the cases studied, namely, acetate on nylon, where a small charge is expected, and a large one found. It is not clear at present whether this anomaly represents a real shortcoming of the correlation scheme, or whether the quantitative considerations have been pushed too far.

We proceed to a detailed consideration of the dependence of transferred charge on various mechanical and ambient conditions.

1. *Rub Type*

In the apparatus used for the study in reference [7], the lower filament was fixed horizontally and the upper filament was moved rectilinearly across it. By varying the azimuths of the filaments, rubs of different types could be obtained. These rubs were identified by giving the azimuthal positions of the bottom and top fibers, respectively. In particular, the symmetric rub, designated 45-45, in which successive portions of the top fiber contacted successive portions of the bottom filament, could be obtained by rotating each filament in opposite directions from parallelism through 45°, so that they were crossed at 90°. Two kinds of asymmetric rub, 0-90 and 90-0, in which either of the filaments could be rubbed in one spot by successive portions of the other, were obtainable in obvious fashion.

(*a*) *Metal–metal.* Since charge flows freely in metals, equilibrium is attained almost instantly. Furthermore, any charge transferred will flow freely along the wire. Hence the charge left on one wire will be independent of area rubbed, and will depend only on the conditions at separation. The net charge will be small, and nearly independent of rub type. We did not investigate metal–metal contacts systematically, but did notice that the charge measured was only a few per cent of that measured with most insulator–metal or insulator–insulator contacts, and that this small quantity did not appear to vary with type of rub.

(*b*) *Metal–insulator.* Since charge flows freely in metals, but only with difficulty in insulators, we should expect that when a large area of insulator is rubbed, a large charge is developed. In an asymmetric rub where one spot on the metal is rubbed by a length of insulator, the charge should be large; in the converse type of rub, the charge should be small; in a symmetric rub the charge should be large. These predictions are substantiated.

(*c*) *Insulator–insulator.* The analysis for this case is similar to that for metal-insulator, provided we consider "metal" replaced by "insulator of higher conductivity," and "insulator" by "insulator of lower conductivity." We shall expect, however, that the demarcation is not so sharp as it is in the previous case. In Figure 9 of reference [7] there is seen to be excellent corroboration of this prediction; for the asymmetric 0-90 rub (in which one spot on insulator of higher conductivity is contacted), the actual length of stroke is 0.707 times the actual length for the symmetric 45-45 rub, and charges are very nearly in this ratio; for the asymmetric 90-0 rub (one spot on insulator of lower conductivity), the charge is small.

There is, however, another finding to be considered in the case of asymmetric rubbing, which concerns the direction of charge transfer as well as the magnitude. The experimental findings can be summarized as follows: (1) When one spot on the material of lower conductivity is rubbed, a small charge is developed, and this charge may have a different sign from the case of symmetric rubbing. (2) When one spot on a filament is rubbed by continually renewing the surface of the *same* material (i.e., 90-0 or 0-90 rubs between filaments of the *same* material), the sample rubbed at one spot becomes positive. We believe that the explanation lies in the local heating, the increased temperature raising the Fermi level of the material rubbed at the same spot.[10]

If this is the correct explanation, then not merely would we find that the material rubbed at one spot by successive portions of this same material becomes positive, but we might expect even that for pairs of different materials with Fermi levels nearly the same in isolation, one level could be shifted enough by heating to reverse the direction of electron transfer from that in a symmetric rub to that in one of the asymmetric rubs. Nylon on aluminum seems to provide such a case, and the explanation seems reasonable; but the same effect is found with nylon on magnesium, and it is hard to believe that the shift could be so great. So this explanation must not be taken too seriously.

[10] It is of course gratuitous to assume that the Fermi level is raised by increasing the temperature. In theory it can shift either way, depending on the details of the atomic field. Perhaps it does, for indeed there are one or two possible exceptions to the finding (2) above.

2. *Length of Stroke*

(*a*) *Metal–metal contacts.* As stated earlier, when charge can flow freely along the filament, the net charge transferred should depend only on the conditions at breaking of contact. Hence there should be negligible effect of length of stroke. This was found to be the case.

(*b*) *Metal–insulator, and* (*c*) *Insulator–insulator contacts.* If the charge is localized in the material of lower conductivity, then the net charge transferred should be proportional to the area contacted on that material. Hence for symmetric rubbing the charge transferred should be proportional to length of stroke, plus a small amount arising from contact alone. Such is found to be the case (*cf.* [7], Figures 5–8). For asymmetric rubbing, when one spot of the material of higher conductivity traverses different spots on the material of lower conductivity, the same result would be expected; in the converse case, the charge transferred would be small, and more or less independent of length of stroke. These expectations are confirmed.

3. *Velocity*

An increase in rubbing velocity would seem to have two main effects. (1) It would decrease time available for charge to flow through the gap upon separation, and for charge to flow away from the point of contact; and (2) it would increase the local temperatures. So far as the first effect is concerned, it is unlikely that it would cause appreciable change in net charge transferred, in view of Harper's findings on the effect of velocity of separation [5]. So far as the second effect is concerned, we suppose that in 45-45 rubs at least the shift in the Fermi levels is small; but degree of approach to equilibrium may be shifted significantly (*cf.* Frenkel [3]), and the conductivity may be increased. So except in cases where a nonempty band overlaps a nonfilled band (*cf.* Figures 2*b*, 3*a*, 3*d*), the charge transferred may be increased by increasing the velocity, provided the frictional heating is enough to raise the temperature appreciably. Moreover, whether or not this overlap occurs, if an insulating material has its conductivity raised much by heating, the net charge transferred may be increased in cases where it would otherwise be cut down to a high degree by reverse flow through the gap upon separation.

When a dependence of charge on velocity exists, we should expect the charge to increase with increasing velocity, but all in all, we should expect this dependence to be slight. These expectations are borne out qualitatively (*cf.* Figures 9–15 of reference [7]), but so little is known quantitatively about the frictional temperature rise and the behavior of the material that we believe it supererogatory to attempt further comparison.

4. *Force*

As stated in Section II, D of the present paper, we have assumed that the true area of contact depends primarily on the normal force, and only secondarily on the macroscopic geometry. In the case of nylon and viscose on polyethylene, as discussed in reference [7], p. 289, there is good evidence in support of this assumption, since the same charge was obtained for the same force produced in two methods involving different macroscopic geometries. So far as the form of dependence of charge on force is concerned, it is seen in Figures 16–22 of reference [7] that as a rule the curves cannot be extrapolated through the origin. Hence we cannot assume a proportionality of true area of contact to normal force, but must make some additional hypotheses as to the true area at light loads. Further work at lighter forces is necessary, but the apparatus used in the study of reference [7] would first have to be refined.

At high forces, the curves flatten off in some cases; it is our opinion that this leveling is the result of atmospheric breakdown.

5. *Tension*

Unless the stress produced in a material by tension causes a shift in the Fermi level which is significant compared with the difference between Fermi levels in the material rubbed, no change in the charge transferred would be expected. For nylon and acetate, at least, no effect was found (reference [7], Figure 23). It is doubtful if any appreciable effect exists for other pairs of substances, except perhaps for the same substance at two different tensions.

6. *Ambient Temperature*

Unless the change in ambient temperature is significant compared with the difference between room temperature and temperature during contact, no effect would be expected so far as charge initially transferred is concerned. The decrease in resistivity with increase in temperature for materials of inter-

mediate conductivity might have an effect in certain cases (*cf.* following section on relative humidity). No extensive study of this question has been made; however, in reference [7] it was noted in a few measurements of charge generated by nylon on polyethylene at 24.5° C. that the charge was not appreciably different from that at 30° C.

7. Relative Humidity

A change in relative humidity produces a change in the equilibrium moisture content of most materials, and hence alters the chemical nature. The Fermi level, of course, would then shift, but we believe that this effect is slight, except possibly at very high regains. In particular we do not believe that it will alter the *order* in a triboelectric series. The data in reference [7], Figures 24 and 25, confirm this conclusion. The net *amount* of charge transferred will be affected of course, primarily because of the change in resistance. At very high humidities the charge can leak along the fiber fast enough to keep much net charge from transferring. The relaxation time for such a process would have to be smaller than 10^{-2} or 10^{-3} sec., the time required for the filament to traverse a distance comparable with the length of contact area. For a substance with a dielectric constant of about 5 and resistivity of about 10^{8} ohm-cm., the relaxation time is around 5×10^{-3} sec. Thus at relative humidities such that resistivity is 10^{9} ohm-cm. or less, the net charge transferred should become very small. The experimental results substantiate this picture in part, but certainly the results for polyethylene are anomalous, unless the surface resistivity is much lower than expected.

It would be consistent with the over-all theoretical picture for some materials to exhibit a maximum in charge transferred as a function of relative humidity. The maximum would occur when the conductivity is high enough that flow of charge away from the contacting area decreases the reverse flow through the gap during separation, but not so high that charge could race along with the contacting area. The data in reference [7] hint at the existence of a maximum, but certainly would have to be extended to verify it.

IV. Conclusions

The theory as described in the previous sections appears capable of providing a semiquantitative explanation of the principal phenomena in static electrification, provided that contact is severe enough that the surface layers are penetrated, and that electrical breakdown does not occur. It appears that the phenomena can be explained without the use of surface states, though it is entirely possible that they do exist, and even control the charging process. So far as localized hot spots are concerned, it is exceedingly likely that they do exist in most rubbing contacts, and that in some cases a large transfer of charge is due to them, as proposed by Frenkel [3]. But on our picture with certain band structures large transfer may occur without heating. A clear choice among the various theories will be possible only with additional data.

V. Acknowledgments

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A Study of Blended Woolen Structures

Part III: Amplification and Extended Application of the Theory of Blend Statistics[1]

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Abstract

At the time of publication of Parts I and II [1, 2], the mathematical approach to establishment of criteria for judging blend distribution had been governed by two factors. First, the experimental procedures involved microscopic examination and counting of fiber sections at yarn cross sections. Second, the materials under examination were stock-blended woolen yarns where the manifest objective of the fiber mixing processes was to achieve random intermingling.

It was desired to extend the appropriate probability theory considerations to a more general level. This has now been accomplished, and suitable statistical criteria have been derived for use with experimental methods of measuring blend composition which involve gravimetric analysis of finite lengths of yarn, roving, or sliver. To this extent the utility of the earlier work has been considerably expanded.

Beyond this, however, the question has been examined as to whether or not the same basic criteria of blend uniformity apply to worsted-type yarns where blending is accomplished by doubling of slivers. It is shown that such methods can be expected to yield no better than a random blended yarn and therefore are subject to the same limiting ideal.

I. Introduction

In Part I [1] of this series, use was made of probability theory to derive several statistical criteria for the ideal random blended yarn. The practical application of these formulations was illustrated for a battery of wool-nylon and wool-viscose yarns, spun on the woolen system.

The formulas developed and applied in this foregoing work were adapted particularly to an experimental method that entailed counting numbers of fiber cross sections at yarn cross sections. This method provided information on longitudinal, radial, and rotational distribution of both blend and total weight. For determination of simple longitudinal blend distribution, however, the practice of embedding and sectioning yarns, and then counting fibers per cross section may become inordinately tedious. It is, therefore, important to develop appropriate criteria for the ideal random blended yarn for use with an experimental technique which involves weighing finite lengths of yarn and then determining the weight per cent composition by suitable chemical or gravimetric means.

In the development of the statistical theory, it was understood that the ultimate objective of any stock-blending process was to achieve completely random intermingling of the blend components. Uniformity of distribution was ruled out as a practical goal to be achieved by present-day techniques. The question arises as to whether the same limitations apply to top-blending or blending of slivers as would apply to stock-blending. This is shown to be the case, and blended yarns of all sorts are therefore amenable to analysis by the present methods.

The need for a general method of evaluating blend regularity in worsted as well as woolen-type yarns is evident from the comment: "Many empirical trials have been carried out to determine the effect of a varied number of operations on the adequacy of mixing, and most of them indicate the normal drawing set is more than adequate. Since, however, there is no easy numerical method of estimating the degree of mixing obtained, work in this field has been less satisfactory than that done on

[1] Part I appeared in Textile Research Journal **25**, 743 (1955); Part II, **25**, 902 (1955). Correction **25**, 968 (1955). This work is being conducted with the joint support of the Office of Naval Research and the Bureau of Supplies and Accounts, U. S. Navy.

irregularity of thickness" [11]. Evidence is given in the latter sections of this report that the normal drawing set may not be adequate to achieve the ultimate in random blending.

II. Gravimetric Determination of Blend

In the derivations pertaining to blend analysis by fiber count at cross sections, it was shown that the fiber staple lengths do not influence the theoretical values of CV of blend, clustering coefficient, or Index of Blend Irregularity so long as the yarn is sampled at distances separated by over one fiber length. In measuring blend variability among yarn samples of finite length, however, it is necessary to take account of fiber mean staple lengths. Moreover, it should be understood that the CV of per cent composition, as determined by this method, will vary with the length of yarn sample measured as well as average yarn denier, and these must both be taken into account.

One anticipated experimental procedure is somewhat as follows.

Cut from a yarn a reasonably large number of samples, each of length L and each separated from any other by more than one fiber staple length. Determine the weight G of each. By suitable chemical means, find the weight of one component in each of the samples taken. Calculate the average and the individual values of per cent blend composition found in the several samples of length L. It is desired now to compare the experimental values of blend thus found with the variability that can be expected if the yarn is composed of a completely random blend.

Two criteria may be employed. They yield essentially the same information, and it may be merely a matter of convenience which to use in any particular case.

III. The Coefficient of Variation of Blend

In Part II [2], the CV of per cent blend composition by weight for an ideal random blended yarn was applied for the case where sample lengths were infinitely short, i.e., yarn cross sections. It can be shown (see Appendix) that the equations given in Parts I and II were special cases of the more general equations:

$$CV\% \text{ weight of wool among samples of length } L = 100\sqrt{\frac{\alpha}{\omega}\frac{1}{\bar{D}}\left(\frac{\alpha\bar{d}_w}{1+L/l_w}+\frac{\omega\bar{d}_a}{1+L/l_a}\right)(1+X)} \quad (1a)$$

$$CV\% \text{ weight of nonwool among samples of length } L = 100\sqrt{\frac{\omega}{\alpha}\frac{1}{\bar{D}}\left(\frac{\alpha\bar{d}_w}{1+L/l_w}+\frac{\omega\bar{d}_a}{1+L/l_a}\right)(1+X)} \quad (1b)$$

where

ω = weight fraction of wool in the blend, found average
$\alpha = 1-\omega$ = weight fraction of nonwool in blend, found average
$\bar{d}_w$ = average denier of wool fibers
$\bar{d}_a$ = average denier of nonwool fibers
$\bar{D}$ = average denier of the yarn
l_w = mean staple length of wool fibers
l_a = mean staple length of nonwool fibers
L = length of individual yarn samples taken for test
$(1+X)$ = a modifying factor that may generally be ignored or taken as about 1.02

This equation would be used as follows. A large length of, say, a wool-nylon yarn has cut from it about 20 samples each of length L. The lengths to be taken will be essentially a matter of convenience, but something in the order of 1 to 10 times the average staple length might be recommended. Each sample is then subjected to chemical treatment which dissolves out one of the fiber components (see for example [10]). The weight of the undissolved residues from each sample is determined, and from this the values of weight per cent blend composition in each of the 20-odd samples of length L is calculated. The mean blend composition is found as is the CV of per cent blend composition among the samples. Another gravimetric analytic method employing specific volume measurements suggested by Preston might also be of interest here [7].[2]

Now, suppose that the average content was found to be 45% wool. The average denier of the yarn should be known or can be directly calculated from the original weights of samples all of length L and suppose that this turned out to be 500. Assume that the yarn had been spun of 3-den., 3-in. staple nylon and 72's 4-in. staple length wool. Say that the yarn length L taken for testing had been 12 in. Therefore

$\omega = 0.45$
$\alpha = 0.55$
$\bar{D} = 500$
$\bar{d}_w = 3.5$ (for 72's wool)
$\bar{d}_a = 3.0$

[2] Since the preparation of this manuscript, a very efficient adaption of this method has been developed. The technique is the subject of a subsequent report.

$$l_w = 4''$$
$$l_a = 3''$$
$$L = 12''$$
$$\text{Let } 1+X = 1.02$$

The theoretical ideal random blend would then yield a CV of per cent weight of wool

$$100\sqrt{\frac{0.55}{0.45}\frac{1}{500}\left(0.55\frac{3.5}{1+12/4}+0.45\frac{3.0}{1+12/3}\right)} \quad (1.02)$$

$$CV\%\text{ wool} = 4.3\%$$

The actual CV will be found from individual experimental values of weight composition of each sample. Comparison of the actual value with the theoretical value should be made on a quotient basis rather than by absolute differences. Thus, if the actual CV is found to be 6%, the real yarn is to be considered about 1.4 times as variable in blend composition as would have been expected for the theoretical ideal random blend. The mere fact that the CV values are both small should not be allowed to mislead the examiner into believing that the effect is slight. Indeed, if yarn samples 20 yd. long had been taken, variability of blend might have appeared to be entirely lacking for a yarn that was poorly blended.

IV. The Index of Blend Irregularity

In Part I, a somewhat different mathematical attack on the problem of evaluating blend irregularity led to the derivation of a dimensionless Index of Blend Irregularity. Here again, as for the CV, the form given was for the case of counting fibers at cross sections.

$$IBI = \sqrt{\frac{1}{n}\sum\frac{(pT_i - W_i)^2}{T_i pq}} \quad (2)$$

In this case a reasonably large number of yarn cross sections, say 20, are made and the number of each kind of fiber counted in each cross section, as well as the total at each section. In Equation 2

n = number of cross sections counted
p = number fraction of wool fiber sections found from the entire count in all sections
$q = 1 - p$ = number fraction of nonwool fibers sections found from the entire count in all sections
T_i = total numbers of both kinds of fibers found at individual sections
W_i = number of wool fibers found at the individual sections

The index so found yields directly a measure of departure from blend randomness with 0 being perfect uniformity, 1 representing ideal randomness and the excess over 1 being a measure of incomplete randomization.

For the case where the blend composition is determined gravimetrically, according to some technique resembling that described in the previous section, the index can also be calculated. It can be shown that the equivalent statistical evaluation of a yarn will result from

$$IBI = \sqrt{\frac{1}{n}\sum\frac{(g_i - \omega G_i)^2}{G_i}} \times \frac{30}{\sqrt{\alpha\omega L\left(\frac{\bar{d}_w\alpha l_w}{L+l_w}+\frac{\bar{d}_a\omega l_a}{L+l_a}\right)}} \quad (3)$$

This equation has been broken into two terms for convenience in application. The second term is a constant for any particular case and can be readily calculated from available information. The first term contains only two experimental variables: the total weight G_i of each individual yarn sample of length L and the weight of wool g_i in each such sample.

The first term is simply found from the data on individual weights determined by gravimetric analysis. The form of the second term, or constant, given in Equation 3 requires, however, that the individual values of G, and g, be taken in milligrams, and the yarn length L and fiber lengths l_w and l_a used in the constant term are given in centimeters.

V. General Applicability of the Statistical Theory

As has been remarked in the earlier articles of this series, the general approach which leads to Equations 1 through 3 is based on the assumption that all manufacturing processes operate to mingle fibers on a random basis. The criteria are employed to test the ultimate randomness of the blend and hence the efficiency with which the machinery accomplished its required task.

The basic premise involved here would be entirely applicable to cases where stocks are blended at opening in the cotton system or in a mixing picker in the woolen system, for example. In such cases, the obvious intent is to achieve random inter-

mingling of fibers as loose stock. Subsequent doubling operations, if any, are aimed at further randomization of the already partially randomized fiber assemblies.

It may be questioned whether the same criteria of blend regularity are logically applicable to cases where blending is accomplished entirely by doubling as for example to top blending in the worsted system. One might, at first, presume that blends more uniform than random might be achieved by such methods.

Cox [3], however, in the development of a statistical theory of blending by doubling for worsted-type yarns arrives at quantitative results compatible with those of the current work.

Hodgson [5] summarizes the general experience of spinners of blended worsted yarns:

"1. The most even distribution or mixture results when 50/50 blends are made.

2. As the percentage of either component is increased or decreased from 50%, the degree of efficiency of blending becomes progressively worse. (Author's note: the word 'efficiency' should probably be interpreted as 'evenness.')

3. As the number of fibers in the cross section of the sliver, slubbing, or yarn, is decreased, the more irregular or streaky does the mixture appear.

4. There appears to be a limit to the degree of blending that can be achieved, and no amount of additional processing involving doubling and drafting will improve the blending."

These observations are qualitatively identical with the predictions of the statistical theory employed in the present work. Hodgson finds similar concordance between such mill experience and the qualitative implications of normal probability theory. Due note of his contribution was not made in earlier reports in this series only because our own manuscripts had already been prepared prior to the appearance of Hodgson's observations in the literature.

It would appear that the weight of evidence is in favor of applying the statistics of the random blend to worsted yarns as well as woolen. However, a somewhat more rigorous proof would not be amiss. Therefore, consider the simplest case.

For example, a strand of nylon is laid down side by side with a strand of wool. Assume that each strand contains, on the average, the same number of fibers per cross section, and each has the same variability of fiber number per cross section. Ignoring now the fact that there is no lateral intermingling of fibers, the question is: what will this 2-ply arrangement exhibit in the way of longitudinal blend uniformity?

If such a 2-ply sliver or top starts out more uniform in blend regularity than could be achieved by completely random sliver, then it is altogether conceivable that yarn produced therefrom might also exhibit blend regularity better than random. In such a case, this yarn would be found to have a CV of per cent blend composition lower than that predicted by Equation 1 and an IBI as found by use of Equation 3 of less than 1.0. Going further, if the top blending system can theoretically produce a sliver strand superior in blend regularity than random, but final yarn is found from practice to have an IBI greater than 1.0, then there is good reason to scrutinize the processing system carefully to ascertain at which stages the longitudinal blend regularity has been degraded.

On the other hand, if the 2-ply strand itself is theoretically incapable of presenting anything better than a longitudinally random blend, then yarns that eventually emerge from this sliver cannot be expected to be superior in blend uniformity to the random ideal. This is apparent when it is realized that further doublings of the 2-ply strand would at best simply represent randomly mixing together samples of a blend which is no more uniform than random to start. The ultimate yarn could not then be expected to exhibit a CV of blend smaller than that given by Equation 1, nor an IBI below 1.0, and these become valid criteria for top-blended yarns as well as the other types.

It is desired, therefore, to calculate first the theoretical variation in blend composition from one spot to another along the length of a sliver made up by doubling two strands each composed of a different fiber type. Then the blend variation is to be calculated for a sliver equal in thickness to the doubled sliver, but composed of a randomly distributed mixture of the two kinds of fibers, for example, where mixing has occurred before carding. Finally the two results are to be compared.

a. Blend Variation in the 2-Ply Sliver

According to classical formulations [6], the variability of fiber numbers per cross section in a sliver will be

$$\sigma_T = \sqrt{\bar{T}} \tag{4}$$

The actual variability has almost universally been found to exceed $\sqrt{\bar{T}}$. Without attempting here to define the various possible causes that magnify the variation in total number of fibers per cross section, it will be assumed that the slivers to be doubled are each governed by

$$\sigma_T = k\sqrt{\bar{T}}$$

where k is a correction factor related to yarn weight irregularity.

Take a nylon sliver containing $\bar{N}$ average number of fibers per cross section and lay it side by side with a woolen sliver containing $\bar{W}$ average number of wool fibers per cross section. The combined, or 2-ply, sliver will therefore contain $\bar{W} + \bar{N} = \bar{T}$ average total number of fibers per cross section. The variabilities of fiber numbers per cross section for each of the two original homogeneous slivers will be taken as

$$\sigma_N = k\sqrt{\bar{N}} \tag{5}$$

$$\sigma_W = k\sqrt{\bar{W}} \tag{5a}$$

and the variability of total number of fibers per cross section in the 2-ply will be

$$\sigma_T = \sqrt{\sigma^2_W + \sigma^2_N} \tag{6}$$

$$\sigma_T = k\sqrt{(\bar{W} + \bar{N})} = k\sqrt{\bar{T}} \tag{6a}$$

The principal hypothesis of the 2-ply assemblage is that the slivers are randomly juxtaposed next to each other, so that a given number of wool (say W_i) fibers can be associated with any value of nylon fibers. The values W_i and N_i found at the same sections of the 2-ply are therefore entirely uncorrelated, but this is not true of the relationship between W_i and the sum of W_i and N_i. Thus, the value of T will tend to go up or down as W varies up or down. It will be necessary shortly to know the correlation coefficient for the relationship between W and T. Therefore this is now calculated.

Consider Figure 1. Here values of W are plotted against values of T (i.e., $W + N$), as found at individual cross sections.

The variation of any value of T, where W is fixed, will be due only to the possible variation of N. Such being the case, the standard error of estimate of T, for given values of W, will be

$$S_{eT} = \sigma_N = k\sqrt{\bar{N}} \tag{7}$$

The total variation of T has already been given in Equation 6. The correlation coefficient r will be found from

$$r^2 = 1 - \frac{S^2_{eT}}{\sigma^2_T} \tag{8}$$

and is therefore

$$r = \sqrt{1 - \frac{\bar{N}}{\bar{T}}} \tag{9}$$

In Equation 9, $\bar{N}/\bar{T}$ is simply the average number proportion of nylon fibers in the blend. In accordance with previous terminology,

$$\frac{\bar{N}}{\bar{T}} = q = 1 - p$$

and therefore

$$r = \sqrt{p} \tag{10}$$

where p = average number proportion of wool fibers per section.

The variation in blend proportion in the 2-ply strand is the variation in the ratio W_i/T_i, and will

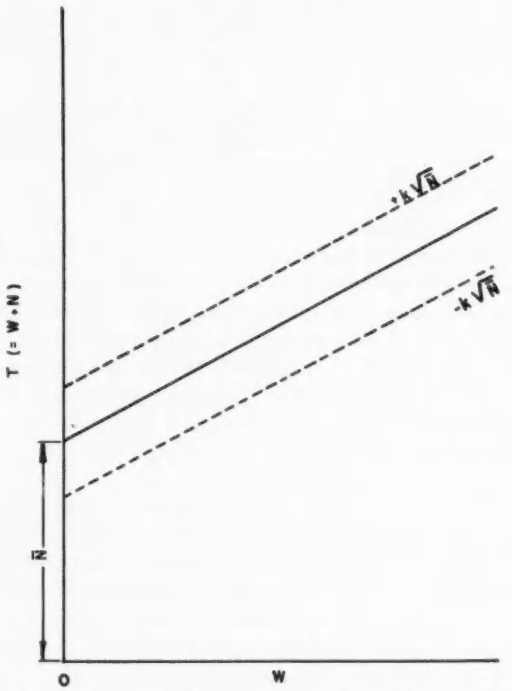

Fig. 1. Illustration of regression of T on W with standard error of estimate of T indicated by $k\sqrt{\bar{N}}$.

be found from

$$CV^2_{W/T} = CV^2_W + CV^2_T - 2rCV_WCV_T$$
$$= \frac{k^2}{\bar{W}} + \frac{k^2}{\bar{T}} - 2\sqrt{p\frac{k^2}{\bar{W}}\frac{k^2}{\bar{T}}}$$
$$= k^2\left(\frac{1+p-2p}{p\bar{T}}\right)$$

$$CV_{W/T} = k\sqrt{q/p\bar{T}} \quad (11)$$

b. Blend Variation in the Random Sliver

Consider a sliver resulting from random intermingling of fiber stock, containing on the average $\bar{T}$ total number of fibers. In the sliver there will be found $p\bar{T}$ average number of wool fibers per section and $q\bar{T}$ number of nylon fibers. The variability of number of wools or nylons for cross sections containing T_i total number of fibers will be found from

$$S_{Wi} = \sqrt{T_i pq} \quad (12)$$

$$S_{Ni} = \sqrt{T_i pq} \quad (12a)$$

where

$p = \frac{\bar{W}}{\bar{T}}$ = average number proportion of wool

$q = 1 - p$ = average number proportion of nylon

T_i = any fixed value of total fibers

W_i = the variable number of wools associated with T_i

N_i = the variable number of nylons associated with T_i

For sections having the same value of T_i, the variation of blend will be due only to the variation of W_i, and therefore the variability of W_i/T_i will be

σ_i (blend proportion)

$$= \frac{S_{wi}}{T_i} = \frac{\sqrt{(T_i pq)}}{T_i} = \sqrt{pq/T_i} \quad (13)$$

The over-all variation of blend proportion for all sections with varying values of T will be the root mean square, or

$\bar{\sigma}$ (blend proportion)

$$= \sqrt{f_1\left(\frac{pq}{T_1}\right) + f_2\left(\frac{pq}{T_2}\right) + \cdots/\Sigma f} \quad (14)$$

$$= \sqrt{\frac{pq}{\Sigma f}\Sigma\frac{f_i}{T_i}} \quad (15)$$

where f_i = frequency of occurrence of sections containing T_i total numbers.

A plot of f versus T will, of course, be a frequency distribution curve representing the thickness variability of the sliver. If the sliver had perfect uniformity in weight along its length, and therefore no variability of T,

$$\frac{1}{\Sigma f}\Sigma\frac{f_i}{T_i} = \frac{1}{\bar{T}}$$

But there is known to be a weight variation along the sliver and hence,

$$\frac{1}{\Sigma f}\Sigma\frac{f_i}{T_i} = \frac{1}{\bar{T}}(1+X)^* \quad (16)$$

where $(1 + X)$ = a factor depending on the dispersion of T. From Equations 15 and 16, therefore

$$CV_{W/T} = \sqrt{\frac{pq}{\bar{T}}(1+X)} \div \frac{\bar{W}}{\bar{T}}$$
$$= \sqrt{\frac{q}{p\bar{T}}(1+X)} \quad (17)$$

c. Comparison of Theoretical Blend CV's

For the doubled strand,

$$CV_{W/T} = k\sqrt{q/p\bar{T}} \quad (11)$$

For the stock-blended, ideal random blend,

$$CV_{W/T} = \sqrt{(1+X)}\sqrt{q/p\bar{T}} \quad (17)$$

Obviously, the comparison here is between k and $\sqrt{1+X}$. Both these terms are directly dependent on the over-all weight variability of the slivers. Assuming that the two sorts of slivers in question suffer from the same degree of weight irregularity, it can be shown (see Appendix) that to a very close approximation:

$$\frac{1}{\Sigma f}\Sigma\frac{f_i}{T_i} = \frac{1}{\bar{T}} + \frac{\sigma^2_T}{\bar{T}^3} \quad (18)$$

Letting $\sigma_T = k\sqrt{\bar{T}}$, as previously, then

$$\frac{1}{\Sigma f}\Sigma\frac{f_i}{T_i} = \frac{1}{\bar{T}}\left(1 + \frac{k^2}{\bar{T}}\right) \quad (19)$$

From Equation 16, therefore

$$X = \frac{k^2}{\bar{T}} \quad (20)$$

Plots of $(1 + X)$ versus k are given in Figure 2 for several values of $\bar{T}$.

Values of k of about 1.15 are given as reasonable by Cox [3] for worsted yarns, and values of at least

* See Appendix II.

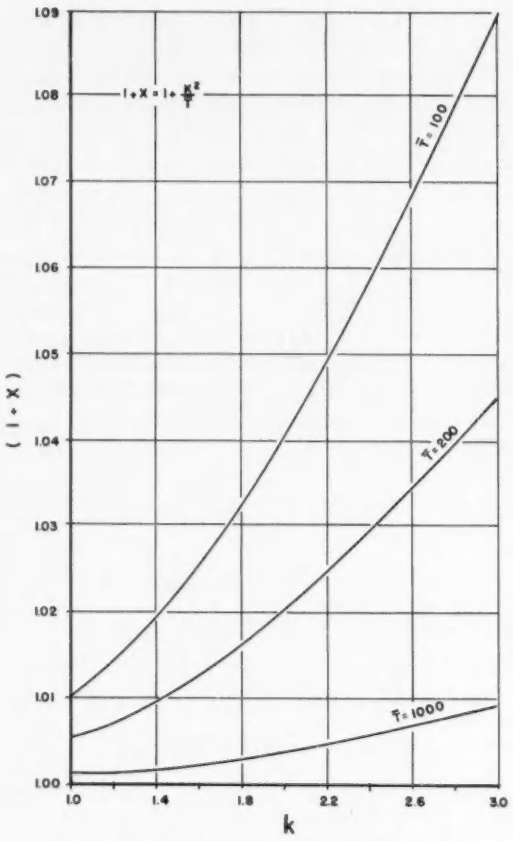

Fig. 2. Relationship between weight irregularity factors k and $(1 + X)$.

2–3 for k can be derived from information given by Martindale [6] for botany slivers using 64's wool. Data from Part I [1] yield values for k of about 2 for woolen yarns. Both Hasler and Honegger [4] and Temmerman and Hermanne [8] give values for k of about 1.5–2.5 for cotton yarns and as much as 10 for drawn and combed slivers.

It will be seen that in realistic cases for yarns where $k = 1.15$, $(1 + X) \doteq 1.01$, where $k = 2.5$, $(1 + X) \doteq 1.05$. In slivers, where $k = 2$, $(1 + X) = 1.005$, and even when k goes to 10, $(1 + X)$ is only 1.10.

Consequently, one can expect in the 2-ply sliver a blend irregularity at least double and as much as about 10 times greater than what would be expected of a completely random sliver. Repeated doublings and drawings would be required not only to effect lateral intermingling, but to reduce the longitudinal blend irregularity. Cox [3] gives theoretical proof that more doublings are required to achieve the lateral intermingling. Townend and Griffin [9], however, point out some very practical difficulties in achieving longitudinal randomization of stocks fed to gilling.

The ideal random blend seems to be the limit of longitudinal uniformity not only for woolen (or stock-blended) systems in general but for worsted (or sliver and top-blended) systems as well. The applicability of Equations 1 through 3 is therefore completely general for analysis of blended yarns from any spinning system provided that the average blend concentration of the minor component is in excess of about 10%. For the use of Equation 2, there is the additional proviso that the average number of fibers per cross section should exceed about 50. This restriction might ordinarily apply to cotton yarns as fine as about 80's or finer and to worsted yarns as fine as about 50's or finer. In such instances, Equations 1 and 3 could still be employed if samples about double the average fiber staple length were the minimum length taken for analysis.

VI. Conclusions

1. Statistical methods previously developed for evaluation of blend randomness by a microscopical technique have been amplified.

2. A general method for evaluating blend uniformity is given for use with experimental techniques which involves gravimetric analysis of finite lengths of yarn.

3. It is shown that the uniformity of the theoretical ideal random blend depends on seven independent variables: (*a*) average yarn weight, (*b*) average blend composition, (*c*) fiber denier of one component, (*d*) fiber denier of the other component, (*e*) staple length of one component, (*f*) staple length of the other component, and (*g*) to a minor extent, the over-all weight irregularity of the yarn.

4. The CV of % weight composition not only is dependent on the foregoing, but also will be influenced by the lengths of sample taken for analysis.

5. It is shown that it is impossible to achieve a more uniform blend by doubling than could be expected from complete randomness.

6. In view of 5, the statistical criteria for the ideal random blend are applicable for cases of top and sliver blending as well as for stock-blending.

Appendix I

Analysis of Blend Irregularity for Use with Gravimetric Data

The following outline summarizes the steps which will be used in the formal derivation.

1. Expressions will be formulated for the average number and effective staple length (as later defined) of wool and nonwool fibers in a given length of yarn.
2. Relationships will be established among fiber denier, fiber length, number of fibers, length of yarn section, and section weight.
3. Previously developed formulas for distribution of numbers of fibers are applied to the above.
4. The distribution of numbers of fibers so found is expressed in terms of weight parameters.
5. The distribution in terms of weight parameters is normalized to one of number deviations of wool fibers with fixed total weight of yarn section.
6. These number deviations are next converted to weight deviations of wool, following which they are expressed as CV of % weight of wool and finally as Index of Blend Irregularity.

At a yarn cross section there will be on the average, by definition, $\bar{W}$ wool fibers. In traversing the yarn lengthwise, there will be encountered on the average $\bar{W}$ new fiber ends in a length of yarn equal to the fiber staple length; thus for a random distribution of fibers the number will be $\bar{W}\ (L/l_w)$ new fiber ends in a length of yarn L, where l_w is the fiber staple length. Then in a yarn section of length L, there will be

$$W_L = \bar{W}\left(1 + \frac{L}{l_w}\right)$$

whole or fractional wool fibers, where l_w is the wool staple length, and

$$A_L = \bar{A}\left(1 + \frac{L}{l_a}\right)$$

whole or fractional nonwool fibers, where l_a is the staple length of the nonwool fibers and $\bar{A}$ is the average number of nonwool fibers per cross section.

The total length of the wool fibers will be $\bar{W}L$. Thus

$$l_{wL} = \frac{\bar{W}L}{\bar{W}\left(1 + \frac{L}{l_w}\right)} = \frac{l_w L}{l_w + L}$$

where l_{wL} is the average length of a wool fiber fragment in the section of length L.

Similarly, for the average length of nonwool fibers,

$$l_{aL} = \frac{l_a L}{l_a + L}$$

The total weight G milligrams of a yarn section of length L cm may be expressed as

$$900G = W_L \bar{d}_w l_{wL} + A_L \bar{d}_a l_{aL} \qquad \text{(I)}$$

where $\bar{d}_w$ and $\bar{d}_a$ are the average deniers of the wool and nonwool fibers, respectively.

$$W_L + A_L = T_L \qquad \text{(II)}$$

where T_L is the total number of fiber fragments in a section of length L.

Thus, for a given weight of yarn section there is a unique relation between W_L and T_L. By defining $\beta = \bar{d}_w/\bar{d}_a$ and combining Equations I and II, there results

$$W_L = \frac{900G\beta - \bar{d}_w l_{aL} T_L}{\bar{d}_w(\beta l_{wL} - l_{aL})} \qquad \text{(III)}$$

$$T_L = \frac{900G\beta - W_L \bar{d}_w(\beta l_{wL} - l_{aL})}{\bar{d}_w l_{aL}} \qquad \text{(IIIa)}$$

Defining ω and α as the weight fraction and p' and q' as the number fraction of wool and nonwool fiber fragments, respectively, and γ as ω/α, the following relations hold:

$$900\omega G = \bar{d}_w l_{wL} \bar{W}_L$$

and

$$900\alpha G = \bar{d}_a l_{aL} \bar{A}_L$$

where $\bar{W}_L$ and $\bar{A}_L$ represent the average values of W_L and A_L, respectively. Dividing,

$$\frac{\omega}{\alpha} = \frac{\bar{d}_w l_{wL} \bar{W}_L}{\bar{d}_a l_{aL} \bar{A}_L}$$

or

$$\gamma = \beta \frac{l_{wL}}{l_{aL}} \cdot \frac{p'}{q'}$$

Substituting the identity $q' = 1 - p'$,

$$p' = \frac{\gamma l_{aL}}{\beta l_{wL} + \gamma l_{aL}} \qquad \text{(IV}a\text{)}$$

and

$$q' = \frac{\beta l_{wL}}{\beta l_{wL} + \gamma l_{aL}} \qquad \text{(IV}b\text{)}$$

By definition,

$$p'\bar{T}_L = \bar{W}_L$$

where the barred values are average values. Thus

when

$$W_L = p'T_L$$

and

$$W_L = \bar{W}_L \qquad \text{(V)}$$

then

$$T_L = \bar{T}_L$$

From Equations III through V

$$\bar{T}_L = \frac{900G(\gamma l_{aL} + \beta l_{wL})}{\bar{d}_w l_{wL} l_{aL}(1 + \gamma)} \qquad \text{(VI)}$$

What is wanted now is the distribution of W_L about $\bar{W}_L$, or in normalized form

$$\Delta\bar{n} = \frac{W_L - \bar{W}_L}{\sigma_w} \qquad \text{(VII)}$$

It is known that the standard deviation of number of wool fibers in a section of T_L total fibers is given by $\sqrt{T_L p'q'}$.

Thus for fixed T_L, $\bar{W}_L = p'T_L$ and

$$\Delta n = \frac{W_L - p'T_L}{\sqrt{T_L p'q'}} \qquad \text{(VIII)}$$

The same expression holds if T_L is a variable, but in order to use it, T_L must be expressed in terms of the independent variables as in Equation III*a*. Such calculations have been performed and the distribution function plotted for a number of cases. These results show that although a slight skew exists in most cases, all practical cases may be considered symmetrical. This is equivalent to saying that in the numerator of Equation VIII the value of T_L from Equation III*a* will be used while in the denominator T_L will be taken equal to T_L from Equation VI. Thus from Equations III*a* and VIII,

$$\Delta n = \frac{\dfrac{\beta l_{wL}(1 + \gamma)}{\beta l_{wL} + \gamma l_{aL}}\left[W_L - \dfrac{900G\gamma}{\bar{d}_w l_{wL}(1 + \gamma)}\right]}{\sqrt{T_L p'q'}} \qquad \text{(IX)}$$

Equating Equation VII with Equation IX,

$$\sigma_w = \sqrt{T_L p'q'}\,\frac{(\beta l_{wL} + \gamma l_{aL})}{\beta l_{wL}(1 + \gamma)} \qquad \text{(X)}$$

and

$$W_L = \frac{900G\gamma}{\bar{d}_w l_{wL}(1 + \gamma)} \qquad \text{(XI)}$$

That this expression is reasonable may be checked by using $\bar{W}_L = p'\bar{T}_L$ and substituting the values from Equations IV*a* and VI. This, in fact, is an alternative means of arriving at Equations X and XI.

To convert Equation X from standard deviation by number to standard deviation of wool fibers by weight in milligrams, multiply Equation X by $\bar{d}_w l_{wL}/900$. Then

$$(\sigma_w)_{\text{wt.}} = \sqrt{\bar{T}_L p'q'}\,\frac{(\beta l_{wL} + \gamma l_{aL})\bar{d}_w l_{wL}}{\beta l_{wL}(1 + \gamma)(900)}$$

which after substitution in terms of the basic quantities becomes

$$(\sigma_w)_{\text{wt.}} = \frac{1}{30}\sqrt{\alpha\omega GL\left(\frac{\omega\bar{d}_a l_a}{l_a + L} + \frac{\alpha\bar{d}_w l_w}{l_w + L}\right)} \qquad \text{(XII)}$$

The coefficient of variation by weight is

$$(CV_w\%)_{\text{wt.}} = \frac{(\sigma_w)_{\text{wt.}} \times 100}{\bar{W}_L \dfrac{\bar{d}_w l_{wL}}{900}}$$

which after substitution becomes

$$(CV_w\%)_{\text{wt.}} = 3.33\sqrt{\frac{\alpha L}{\omega\bar{G}}\left(\frac{\omega\bar{d}_a l_a}{l_a + L} + \frac{\alpha\bar{d}_w l_w}{l_w + L}\right)} \qquad \text{(XIII}a\text{)}$$

or

$$(CV_w\%)_{\text{wt.}} = 100\sqrt{\frac{\alpha}{\omega\bar{D}}\left(\frac{\omega\bar{d}_a l_a}{l_a + L} + \frac{\alpha\bar{d}_w l_w}{l_w + L}\right)} \qquad \text{(XIII}b\text{)}$$

for sections of constant weight G and length L. For sections of constant length but variable G the results are

$$(CV_w\%)_{\text{wt.}} = 3.33\sqrt{\frac{\alpha L}{\omega\bar{G}}\left(\frac{\omega\bar{d}_a l_a}{l_a + L} + \frac{\alpha\bar{d}_w l_w}{l_w + L}\right)} \times \sqrt{1 + X} \qquad \text{(XIII}c\text{)}$$

and

$$(CV_w\%)_{\text{wt.}} = 100\sqrt{\frac{\alpha}{\omega\bar{D}}\left(\frac{\omega\bar{d}_a l_a}{l_a + L} + \frac{\alpha\bar{d}_w l_w}{l_w + L}\right)} \times \sqrt{1 + X} \qquad \text{(XIII}d\text{)}$$

when $\bar{G}$ is the average total weight of sections of length L, $\bar{D}$ is the average denier of sections of length L, and

$$(1 + X) = \left[\frac{1}{n}\sum_{i=1}^{n}\frac{f_i}{T}\right] \div \frac{1}{\bar{T}}$$

as described in the text and derived in Appendix II.

The Index of Blend Irregularity has been defined in terms of numbers [1] as the root mean square of the normalized deviation of measured values at cross sections, the normalizing values being deter-

mined theoretically for a random distribution. The same procedure applies here except that both measured and theoretical values are by weight for sections of finite length.

Then

$$I.B.I. = \sqrt{\frac{1}{n}\sum_{i=1}^{n}\frac{(g_i - \omega G_i)^2}{(\sigma_w)_{\text{wt.}}}}$$

$$= 30\sqrt{\frac{1}{n}\sum_{i=1}^{n}\frac{(g_i - \omega G_i)^2}{\alpha\omega G_i L\left(\dfrac{\omega d_a l_a}{l_a + L} + \dfrac{\alpha d_w l_w}{l_w + L}\right)}} \qquad \text{(XIV)}$$

or expressed in a form easier to use for computation

$$I.B.I. = \sqrt{\frac{1}{n}\sum_{i=1}^{n}\frac{(g_i - \omega G_i)^2}{G_i}} \times \frac{30}{\sqrt{\alpha\omega L\left(\dfrac{\omega d_a l_a}{l_a + L} + \dfrac{\alpha d_w l_w}{l_w + L}\right)}}$$

where G_i is the total weight and g_i the weight of wool in the ith section.

Appendix II

Derivation of the Correction Factor $(1 + X)$

The factor $(1 + X)$ referred to in the text is defined as

$$(1 + X) = \left[\frac{1}{n}\sum_{i=1}^{n}\frac{f_i}{T_i}\right] \div \frac{1}{\bar{T}}$$

$$= \frac{\bar{T}}{n}\sum_{i=1}^{n}\frac{f_i}{T_i}$$

It is evaluated as follows: Substitute the identity

$$T_i = \bar{T}\left[1 + \frac{T_i - \bar{T}}{\bar{T}}\right]$$

Then

$$(1 + X) = \frac{\bar{T}}{n}\sum_{i=1}^{n}\frac{f_i}{\bar{T}\left[1 + \dfrac{T_i - \bar{T}}{\bar{T}}\right]}$$

$$= \frac{1}{n}\sum_{i=1}^{n}\frac{f_i}{\left[1 + \dfrac{T_i - \bar{T}}{\bar{T}}\right]}$$

Expand in a Maclaurin Series:

$$(1 + X) = \frac{1}{n}\Sigma f_i - \frac{1}{n}\Sigma f_i\left[\frac{T_i - \bar{T}}{\bar{T}}\right] + \frac{1}{n}\Sigma f_i\left[\frac{(T_i - \bar{T})^2}{\bar{T}^2}\right] + \cdots$$

$$= \sum_{j=0}^{\infty}\sum_{i=1}^{n}(-1)^j\frac{f_i}{n}\left[\frac{T_i - \bar{T}}{\bar{T}}\right]^j$$

$$= (-1)^j\sum_{j=0}^{\infty}\frac{j\text{th moment of the distribution of } T_i \text{ about } \bar{T}}{\bar{T}^j}$$

The zeroth moment is 1; the first moment, identically zero, and the second moment, the standard deviation squared.

All odd moments vanish identically for symmetrical distributions but for any distribution it can be seen from the double summation that if $(T_i - \bar{T})/\bar{T}$ is much less than unity, $(T_i - \bar{T})/\bar{T}^j$ is very small for higher j's. The only time that $(T_i - \bar{T})/\bar{T}$ is not considerably less than unity is at the tails of the distribution where f_i is vanishingly small. Thus all terms of $j = 3$ or higher will be neglected and there results

$$(1 + X) \doteq 1 + \frac{\sigma_T^2}{\bar{T}^2}$$

or

$$(1 + X) \doteq 1 + \frac{(CV\%)^2}{10^4}$$

Appendix III

A Practical Application of the Theory

The present authors have received a set of data on blend uniformity from the Conservatoire National des Arts & Metiers (French National Ministry of Education). This data constitutes a portion of a report* given by Messrs. Maillard, Roehrich and Amouroux at the International Wool Federation Technical Committee meetings of June 1955 and January 1956.

In this work two blended (worsted) yarns were studied: a 550-den. 50% wool-viscose yarn, and a 440-den. 20% nylon 80% wool yarn. Blend variability was determined in each of these yarns by gravimetric analysis of samples of lengths 5, 10, 50, and 100 cm.

The following fiber information was given

Wool diameter 25.8 μ (approx. 6 den.)
Viscose denier 4
Nylon denier 3
Viscose length 120 mm.
Nylon length 100 mm.

* (Contribution à l'Étude des Melanges de Laines et de Fibres Artificelles et Synthetique en Filature Peignee.)

Lacking better information, it was assumed that the wool fiber length was about 100 mm. The theoretical minimum variability of blend composition was calculated by Equation 1*b*. The ratios of found to predicted *CV*'s were also calculated. Table I presents all these figures.

TABLE I. Comparison of Actual versus Theoretical Minimum Blend Variability Determined by Analysis of Finite Lengths of Yarn

Test length *L* (cm.)	Theoretical min. *CV* (%)	Found *CV* (%)	Ratio found/min.
	50/50 Viscose—Wool Blend		
5	7.8	12.2	1.6
10	6.8	8.5	1.3
50	3.9	7.3	1.8
100	2.9	4.5	1.6
	20/80 Nylon—Wool Blend		
5	14.9	23.7	1.6
10	12.9	23.2	1.8
50	7.4	10.4	1.4
100	5.5	8.1	1.5

Examination of column 3 leads to the observation that the wool-nylon blend is markedly more irregular than the wool-viscose blend. However, the fault does not lie with the type of fiber involved nor to any differences in spinning that may have prevailed in the preparation of the two yarns. The effect observed here is due to the difference in blend proportion in the two yarns. Column 4 indicates that both yarns are equally more irregular than their theoretical minima.

The use of Equation 1, therefore, avoids pitfalls in interpretation of experimental data and minimizes the possibility of drawing unfounded conclusions from otherwise valuable information. Similarly, it points out the necessity for taking due account of sample lengths involved in the testing and makes it possible to compare test results where data was determined from sets of samples differing in length between sets.

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Cotton Quality and Fiber Properties

Part VI: Comparison of a Matched Pair of Raingrown and Irrigated Cottons[1]

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Abstract

Several years ago "irrigated cottons" were discounted under comparable grades and staples of raingrown cottons because of the widespread belief that the processing characteristics of the irrigated cottons were inferior to the normal raingrown cottons. Since Textile Research Institute was engaged in a broad program of relating cotton fiber properties with processing behavior and product quality, it seemed reasonable to include in this program an investigation of whether or not any fundamental differences between raingrown and irrigated cottons exist.

As examples of these two types of cotton, seven bales each of Mississippi raingrown Deltapine 15 cotton and California irrigated Acala 4–42 cotton from the 1951 crop were processed into yarns and fabrics of standard constructions. Conventional fiber tests and tests for single-fiber mechanical properties indicated the cottons were alike in all properties except the energy required to uncrimp the fibers, which was consistently higher for the irrigated cotton. Conventional chemical tests also revealed the irrigated cotton to have a higher wax content, yield a larger ash, and have a lower degree of polymerization.

Definite processing superiority and slightly better product quality were observed for the raingrown cotton in the processing comparison. These differences may be attributed to the lower uncrimping energy for the raingrown cotton or to its different surface characteristics, as manifested by its lower wax and ash contents. From a practical point of view, however, no fundamental, deep-seated difference could be observed between the raingrown and irrigated samples in this test. Most probably, with slight processing organization adjustments, the differences observed in the mechanical processing characteristics of these two cottons would become insignificant.

Introduction

Some years ago "irrigated cotton" produced in California, Arizona, New Mexico, and the Pecos and El Paso valleys of West Texas was discounted under comparable grades and staples of raingrown cotton because of an alleged inferiority in processing behavior and product quality associated with the irrigated cottons [7, 13]. Certainly a part of this mill experience was due to the attempts of mills to employ with irrigated cottons the same processing organization being used with raingrown cotton. Despite the fact that changes in processing procedure are frequently required for conversion from one raingrown cotton to another with properties somewhat different from that customarily processed, the view that irrigated cotton differed in some fundamental respects from raingrown cotton was generally adopted by mill personnel.

While it is true that with increased experience many mills have been able to utilize irrigated cottons most satisfactorily in the production of high quality

[1] This report describes a portion of the work carried out in a broad program relating cotton fiber properties with processing behavior and product quality. A part of this program has been performed under contract with the U.S. Department of Agriculture and authorized by the Research and Marketing Act. The contract was supervised by the Southern Regional Research Laboratory, Southern Utilization Research Branch, Agricultural Research Service. Although the examples used in the present phase of this program were a matched pair of raingrown and irrigated cottons, the conclusions expressed herein by the author regarding the relative merits of raingrown and irrigated cottons, are not necessarily held or endorsed by the U.S. Department of Agriculture.

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cotton products and although irrigated cottons with improved processing characteristics have been developed in the past few years, the question regarding basic differences between raingrown and irrigated cottons has never been satisfactorily answered. Research on this question was therefore undertaken by Textile Research Institute as part of its broad program of investigations on the relations between cotton fiber properties on the one hand and processing behavior and product quality on the other.

It should be emphasized that the present study was limited essentially to a matched pair of typical raingrown and irrigated cottons from the 1951 crop. The conclusions drawn from these cottons may not necessarily apply to all samples of raingrown and irrigated cottons, especially if the samples being compared differ appreciably from each other in conventional fiber properties. Moreover, it is well known that the properties of the same varieties of cotton grown in the same location may vary appreciably from year to year.

It should also be understood in connection with the following discussions that the growing conditions for raingrown and irrigated cottons differ by more than simply the manner in which water is provided to the cotton plant. Irrigated cottons are generally grown in hot arid regions where atmospheric humidities are lower and temperatures are higher than in the raingrown regions. Soil compositions, time of exposure to sunlight, and other factors are also apt to vary. In fact, cottons most suited for raingrown areas might not necessarily grow well in irrigated territory, and *vice versa*.

The Samples

In this research nine- and twelve-bale quantities of Deltapine 15 (Mississippi raingrown) and Acala 4-42 (California irrigated) cottons, respectively, were obtained to compare their fiber properties and processing behavior. (The code words Rainmed and Irrmed, respectively, will be used to identify these two samples in the subsequent discussion.) All bales of the two cotton types were initially matched on the basis of conventional fiber properties of grade, staple, Micronaire fineness, and Pressley flat bundle strength [1]. The bales within each lot were grown in the same field and were selected to be as nearly alike as possible in order to obtain maximum homogeneity.

TABLE I. Conventional Fiber Properties of Rainmed and Irrmed Bale Cotton *

	Micronaire reading, μg./in.	Fibrograph length			Pressley flat bundle strength, 1000 psi, UR, %
		UHM, in.	Mean, in.	UR, %	
Rainmed	4.13	1.13	0.87	77	81.2
	4.37	1.14	0.87	75	82.6
	4.26	1.08	0.81	75	84.3
	4.10	1.12	0.85	76	83.8
	4.22	1.13	0.85	75	83.9
	4.25	1.14	0.88	76	82.7
	4.28	1.11	0.86	77	82.2
	4.34	1.11	0.85	77	85.0
	4.38	1.13	0.87	77	84.3
Average	4.26	1.12	0.86	76	83.3
Irrmed	4.45	1.11	0.84	76	85.3
	4.50	1.11	0.86	77	85.3
	4.54	1.11	0.85	77	85.6
	4.49	1.08	0.84	77	85.6
	4.57	1.12	0.85	76	87.2
	4.58	1.12	0.87	78	87.5
	4.55	1.11	0.87	78	86.1
	4.61	1.10	0.86	78	87.0
	4.41	1.12	0.87	78	85.8
	4.53	1.10	0.85	77	87.5
	4.51	1.11	0.88	79	88.0
	4.58	1.11	0.87	78	85.3
Average	4.53	1.11	0.86	78	86.4

* All cottons were purchased as $1\frac{1}{16}$-in. staple, middling bright.

TABLE II. Fiber Properties of Rainmed and Irrmed Opened Cotton Composites

	Rainmed	Irrmed
Grade	Strict middling	Middling
Staple	$1\frac{1}{16}+$	$1\frac{3}{32}$
Length—fineness array data		
Upper quartile, in.	1.24	1.25
Mean, in.	1.04	1.05
Coefficient of variation, %	28	27
Fineness, μg./in.	4.1	4.2
Maturity, %	84	87
Fibrograph length		
Upper half mean, in.	1.11	1.09
Mean, in.	0.91	0.87
Uniformity ratio, %	82	80
Micronaire reading	4.35	4.72
Causticaire maturity index, %	70.5	74.6
Arealometer		
"A" value	462	441
"D" value	23	19
Convolution angle [11], deg.	13.5	11.6
Reversals per cm. [18]		
Base	25.2	25.4
Tip	30.0	32.9

Fiber property values for the various bales, checked in several laboratories, are shown in Table I. Seven bales of each type were blended in an opening line and rebaled into loose bales for future testing and use. Blended composite samples of each type were taken for laboratory tests.

In the laboratory fiber properties of three types were compared. The first of these involved group fiber properties such as fiber length distribution, flat bundle strengths at various gauge lengths, and fineness measurements. The second type of test involved a comparison of the single-fiber mechanical properties. A third series of comparisons was made of the differences between the samples with respect to their moisture absorption behavior, chemical composition, and degree of polymerization. The results of these various tests may be summarized as follows.

Group Fiber Properties

The results of group-fiber-property measurements are shown in Table II.

It is evident from these data that the cottons were a very close match with respect to those properties measured in routine quality evaluations. The Rainmed cotton had, in general, slightly finer, less mature fibers than the Irrmed. On the other hand, according to the fiber length distributions obtained with the TRI single fiber length tester [17] and shown in Figure 1, the Irrmed sample had slightly more long fibers than the Rainmed. These very small differences, however, would certainly not be picked up by the processor using these cottons.

Pressley flat bundle strengths [14] were determined at three conditions of clamp spacing not only

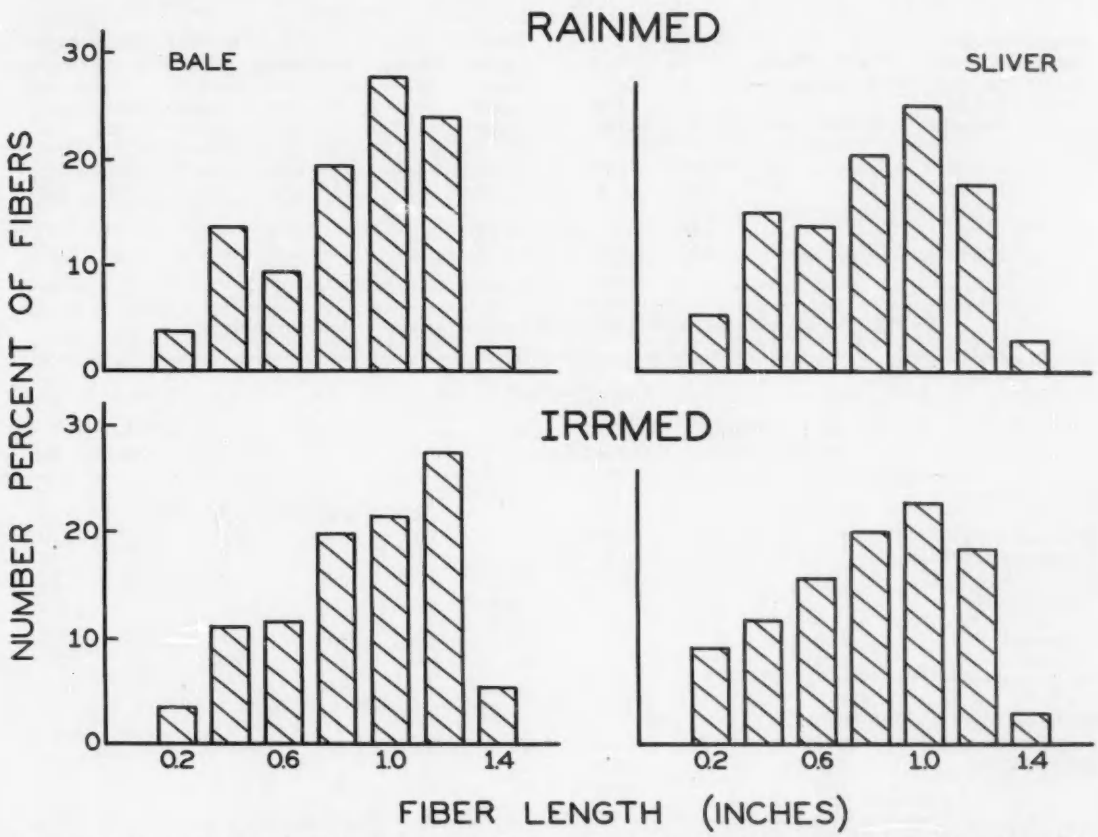

Fig. 1. Fiber length distribution for Rainmed and Irrmed cottons at bale and card sliver stages, obtained by means of the TRI single fiber length tester.

for the blended composite sample but also for samples obtained from nine subsequent processing stations. The results of these tests, summarized in Table III, showed that the cottons differed significantly in the strength loss exhibited with increasing clamp spacing. The Irrmed cotton prior to carding and combing was the stronger cotton at zero millimeter spacing but was the weaker one when the spacing was increased to 5.0 mm. After carding or combing these differences between the cottons were no longer evident. This effect may be related to the difference in fiber crimp since it has been shown that crimp changes with processing through the card and comber and also that the loss in bundle strength from zero to 5-mm. spacing varies with the fiber crimp [16].

Single-Fiber Mechanical Properties

Mechanical properties were determined for single fibers from each cotton type at several processing stations, as outlined in Table IV, using testing procedures previously described [20]. A total of 520 fibers of each cotton were tested following a statistical pattern designed to establish differences between the cottons and trends in fiber property changes with processing.

TABLE III. Flat Bundle Strengths by Cotton, Gauge, and Processing Station

(Mean values in grams/grex representing eight observations)

Gauge, mm.	Cotton	Gin	Opener	Picker	Card	Carded: Drawing sliver	Carded: 1-Hank roving	Combed: Comber	Combed: Drawing sliver	Combed: 2-Hank roving	Combed: 4-Hank roving
0	Irrmed	4.09	4.04	3.96	3.99	4.00	4.10	3.98	4.07	4.04	3.99
	Rainmed	3.83	3.92	3.82	3.86	3.88	3.99	3.77	3.83	3.83	3.92
1.5	Irrmed	2.33	2.12	2.38	2.36	2.41	2.45	2.37	2.47	2.43	2.44
	Rainmed	2.37	2.31	2.37	2.39	2.43	2.40	2.41	2.44	2.42	2.45
5.0	Irrmed	1.58	1.51	1.66	1.59	1.71	1.79	1.69	1.75	1.71	1.77
	Rainmed	1.74	1.66	1.67	1.69	1.71	1.77	1.75	1.78	1.77	1.70

TABLE IV. Mean Values* of Single-Fiber Properties† at Various Processing Stations

(Except for crimp energy, no significant differences between the Rainmed and Irrmed fibers were found for the samples tested.)

	Opened bale	Carded sliver	Finisher drawing sliver: Carded	Finisher drawing sliver: Combed	Roving: Carded	Roving: Combed	Yarn: Carded	Yarn: Combed	Average
Area of cross section $\times 10^{-6}$ cm.^{2}	1.41	1.36	1.38	1.33	1.45	1.38	1.37	1.41	1.39
Breaking load, g.	5.16	4.77	5.61	4.71	5.05	5.26	4.73	5.38	5.09
Breaking elongation, %	7.88	8.48	8.78	8.64	7.89	8.91	7.38	8.20	*
Hookean slope, g./1%	0.654	0.600	0.678	0.582	0.705	0.640	0.898	0.855	‡
Crimp Energy $\times 10^{-3}$ g. cm./cm.									
Rainmed	1.65	1.42	1.01	0.99	1.31	1.04	0.82	0.97	‡
Irrmed	1.98	1.50	1.16	1.06	1.20	1.12	0.93	1.14	‡
Energy to break $\times 10^{-3}$ g. cm./cm.	215	201	244	204	200	230	191	230	‡
Elastic modulus $\times 10^6$ g./cm.^{2}/%	0.470	0.448	0.507	0.439	0.497	0.468	0.688	0.619	‡
Breaking stress $\times 10^6$ g./cm.^{2}	3.68	3.56	4.14	3.56	3.51	3.88	3.49	3.89	3.71

* Combined cotton by station means are based on at least 80 fibers; individual cotton by station means on at least 40 fibers.

† Test methods are described in reference [20].

‡ In the analysis of variance by properties, station effects were found to be significant at the 5% level or better. Isolated pairs of means indicate different populations if they differ by more than the following increments: Breaking elongation, 0.25; Hookean slope, 0.114; Crimp energy, 0.94; Energy to break, 17; and Elastic modulus, 0.183.

It was found that the Rainmed and Irrmed samples were alike with respect to all fiber properties measured except for the energy necessary to uncrimp the fiber. The Irrmed cotton fibers required on the average 8% more energy to uncrimp than the Rainmed ones. This difference appeared to exist throughout the processing from bale cotton to yarn, although the uncrimping energy of both cottons dropped to half of the original value by the time the fibers were made into yarns.

Several other fiber properties for which the two cottons were alike changed during the processing into yarn. Breaking elongation increased during the drawing operations but decreased again during the spinning operation. Hookean slope and elastic modulus of both types of cotton fibers increased abruptly in the spinning operation. No significant changes in breaking load, breaking stress, and energy to break were observed. These observations are in agreement with others previously reported [6, 16].

Chemical Properties

Chemical composition, equilibrium moisture contents, and degree of polymerization indices (intrinsic viscosity) were obtained for both cottons. It was found that the ash and wax contents of the Irrmed sample were definitely higher than the Rainmed. Moisture absorption and desorption behavior appeared to be identical. With respect to intrinsic viscosity, the Rainmed sample was significantly higher than the Irrmed at all stages in processing. These results are summarized in Table V. They confirm in every respect the observations made by Rutherford [13] on similar cottons.

TABLE V. Chemical Properties

	Rainmed	Irrmed
Chemical composition		
Ash in bale	1.07%	1.68%
Ash in roving	1.03	1.45
Wax content	0.58	0.66
Alkali-centrifuge swelling		
Bale	184.9%	184.6%
Roving	199.3	192.3
Moisture content (bale cotton) by		
Absorption	6.97%	6.99%
Desorption	8.11	8.07
Intrinsic viscosity		
Bale cotton	27.3	23.7
Yarn	24.4	22.9
Mercerized print cloth	21.3	17.9
Bleached print cloth	19.8	18.9
Mercerized and bleached print cloth	18.7	16.7

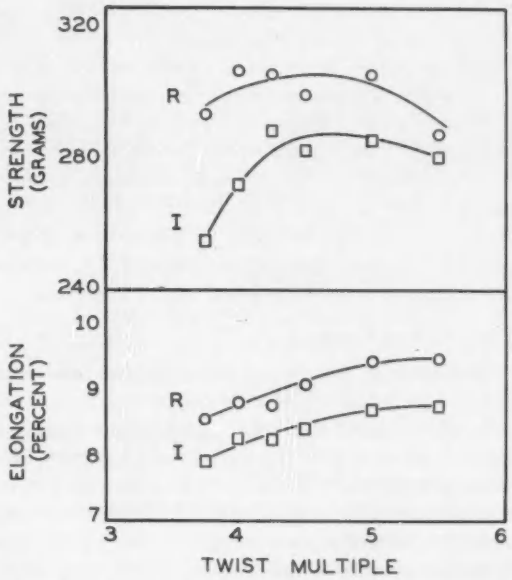

Fig. 2. Effect of twist multiple on single-end strength and elongation of 36's carded yarns of Rainmed (R) and Irrmed (I) cottons.

Small-Scale Spinning Tests

Prior to the pilot-plant processing trials of these cottons a small-scale spinning test was performed at the Clemson Cotton Testing Laboratory of the Cotton Division, Agricultural Marketing Service of the U.S. Department of Agriculture [15]. Yarns of 18's and 36's count were spun, the latter at six twist multiples in the range of 3.75 to 5.5. Skein strengths and single-end strengths and elongations at 2- and 20-in. specimen lengths were determined.

The yarn properties of the Rainmed and Irrmed samples in this test were very much alike, as illustrated in Figure 2. These data show that the Rainmed yarns were somewhat higher in strength and elongation than the Irrmed.

Processing Comparisons

The processing performances of these two cottons were compared in three trials. Two of these involved making 18's and 36's carded yarns and weaving of 48×48 sheeting and 80×80 print cloth, respectively. The third trial included combing at three levels of noils removal and spinning into a 36's yarn for a two-ply sewing thread. Processing experiments were carried out under commercial conditions

in the pilot plants and mills of Dan River Mills and The American Thread Company.

All processing trials were carried out in such a manner that differences between machines in any one processing step would not be mistaken for differences between the cottons. Wherever possible the cottons were processed through the same machines in alternating series to take into account effects due to changes in machine behavior as the processing progressed. A more detailed statement of the processing organization may be found in the Appendix.

Carded Yarn Spinning

Carded yarn processing characteristics are summarized in Table VI.

It will be noted that the Irrmed sample contained a higher percentage of trash than the Rainmed. This difference may be related to the fact that the former was machine-harvested, whereas the latter was hand-picked. Otherwise processing behavior of the two cottons was very similar up to the roving stage. There, at elevated atmospheric temperatures and relative humidities, some difficulty was encountered with the Irrmed lapping up and "eyebrowing" on the cushion top rolls of the roving frame.

Spinning performance of the Rainmed sample was in all yarn constructions superior to that of the Irrmed cotton. In the 18's constructions the spinning end-breakage rates for Irrmed were 53% higher than for the Rainmed; in the 36's construction the rate was 27.5% higher for the Irrmed. The usual differences between warp and fill twists, frames, and checking procedures were observed and taken into account in the statistical design and analysis of these spinning trials [19]. (See also the Appendix.)

It is of interest to note that for both the 18's and 36's carded yarn processing, and in fact, for the combed yarn spinning, the Rainmed roving strengths were greater than those of the corresponding Irrmed rovings (Table VII). This observation that better spinning accompanies higher roving strength confirms similar observations in other processing comparisons [19]. Roving properties thus appear to be a measure of the cooperative behavior of fibers in drafting processes [3], including that occurring in yarn spinning. Since roving strength and stiffness increase with increasing roving twist, it would be of interest to learn if the inferior spinning of the Irrmed cotton could be overcome by increasing its roving twist in each instance sufficiently to bring the roving properties up to the Rainmed values. The answer to this question will have to await future experimentation.

Combed Yarn Spinning

One composite bale each of the Rainmed and Irrmed cottons was processed into combed 36's yarns. Before combing, each lot was divided into three equal parts to be combed with 12%, 16%, and 20% noils removed, respectively. The resulting combed and finisher drawing slivers were kept separate and roved into two roving sizes each: 2.00- and 4.00-hank counts. Thus there were six rovings

TABLE VI. Processing Behavior of Rainmed (R) and Irrmed (I) Cottons

	Carded 18's		Carded 36's		Combed 36's	
	R	I	R	I	R	I
Wastes, %						
Opening	1.16	2.46	—	—	5.00	4.60
Picking	0.64	0.75	0.43	0.84		
Carding	5.61	6.38	6.61	7.46	9.34	10.24
Neps/grain						
Picker lap	12.2	12.1	—	—	—	—
Carded or combed sliver	19.1	19.9	—	—	5.5	4.5
Spinning EDPTSH	13.5	20.8	51.9	66.2	31.4	34.7
Winding breaks, per 1000 quills	7.8	9.4	—	—	—	—
Warping breaks	—	—	51	108	—	—
Evenness, %, by Uster (Linear Integrator)						
Card sliver	3.65	3.53	3.21	3.31	5.24*	5.50*
Breaker drawing sliver	4.36	3.90	4.24	4.30	—	—
Finisher drawing sliver	3.45	3.50	3.79	4.18	4.92*	5.43*
Roving	6.46	6.44	8.28	8.43	9.06*	9.70*

* Quadratic Integrator.

made for each cotton. Processing details may be found in the Appendix.

From all rovings, 36's combed yarns were spun, the 4.00-hank counts being processed by the double-creel process, the 2.00 hank being spun by the single-creel procedure. Differences in the spinning efficiencies of the two cotton types were in the same direction as in the carded yarn tests, but much smaller so that they are significant at a very low confidence level (see Table VI).

From the singles combed yarns in each case a two-plied yarn was made. These yarns were mercerized in a commercial process used for sewing threads. The singles, grey-plied, and mercerized yarns were all evaluated for mechanical properties, evenness, luster, and imperfections.

Weaving of Carded Yarns

In the winding and warping preliminary to weaving, it was observed that more breaks occurred with the Irrmed yarns than with the Rainmed. This difference may be related to the differences in trash content noted earlier, since it was observed in a study of yarn imperfections that the Irrmed combed yarn exhibited more imperfections than the corresponding Rainmed yarn.

In neither the 48 × 48 sheeting nor the 80 × 80 print cloth weaving test was there enough fabric woven or loom hours to give a very reliable indication of weaving efficiency. Nevertheless, it is of interest to note that in weaving the 48 × 48 sheeting the Rainmed yarn showed only two thirds as many breaks as the Irrmed yarn. This observation would seem to be in agreement with the differences in yarn strengths and evenness values shown in Table VII.

Properties of Yarns and Fabrics

Carded yarn evenness, strength, and elongation for the Rainmed cotton were generally similar to the values for Irrmed cotton (Table VII). Such differences as appear in the table were not very great and would very probably have been overlooked in routine mill testing.

In the combed yarn series of experiments, again no important differences between the cottons were observed (Table VII). The Irrmed mercerized yarns were about 5% stronger than the Rainmed, which, of the two, exhibited the higher elongations to break. These differences between cottons were in all cases less than differences attributable to the levels of noils removal.

With respect to fabric properties the cottons were also very similar (Table VIII). The Rainmed fab-

TABLE VII. Properties of Rainmed (R) and Irrmed (I) Fiber Assemblies

	Carded 18's		Carded 36's		(16% Noils) Combed 36's	
	R	I	R	I	R	I
Card sliver						
Strength (g./grex) × 10^6	3206	3091	—	—	3606	3482
Stiffness (g./grex/%) × 10^6	316.4	306.6	—	—	280.4	269.9
Finisher drawing sliver						
Strength (g./grex) × 10^6	931	879	—	—	819	863
Stiffness (g./grex/%) × 10^6	202.4	186.5	—	—	221.4	216.9
Roving						
Strength (g./grex) × 10^4	143	117	420	344	112	95
Stiffness (g./grex/%) × 10^4	241	197	321	293	195	167
Yarn strength						
Single-end break, 10-in. gauge, g./grex, Warp	1.83	1.72	1.68	1.69	1.57	1.66
Filling	1.67	1.65	1.52	1.49	—	—
Skein break factor, CSP (lb.)						
Warp	2476	2441	2101	2085	2301	2360
Filling	2412	2372	—	—	—	—
Yarn evenness, %						
Fielden Walker	11.46	11.77	—	—	12.0	11.0
Uster, Quadratic Integrator	—	—	—	—	17.9	18.0
Uster, Linear Integrator	—	—	18.6	19.0	—	—
Yarn elongation, %						
Warp	8.86	8.06	7.10	6.80	5.25	4.76
Filling	8.17	7.56	6.74	6.18	—	—

rics exhibited somewhat higher elongations than the Irrmed, reflecting differences in yarn and fiber elongation previously mentioned. The rather pronounced superiority in flex abrasion resistance of the Rainmed grey fabrics disappeared with wet processing.

The 80 × 80 bleached and blue-dyed fabrics were tested for reflectance and yellowness, and for color differences. The results of these measurements confirmed visual observations that the Irrmed bleached fabric was slightly more yellow and less reflecting than the Rainmed. Furthermore, both the mercerized and unmercerized Irrmed fabrics, when dyed a blue color, were perceptibly and measurably lighter in shade than the Rainmed counterparts. These differences were well within the limits of what would be considered a commercial color match, however. The extent to which they were caused by the very slight differences in fiber fineness and maturity between these cottons is not known. It is to be noted that in the Goldthwait differential dye test [5] a slight difference in color between these cottons was also observed.

Discussion

The results reported above established a definite processing superiority for the Rainmed sample over the Irrmed: greater roving strength, less lapping in roving, better spinning efficiency, and generally somewhat better yarn quality. Fabric properties were very much the same for the two cottons. Since the differences between the samples were not believed to be predictable from the conventional criteria of cotton quality, a more extensive examination of other properties was made. From this it was found that the Rainmed cotton had (1) slightly fewer long fibers, (2) somewhat less fiber crimp, (3) less wax and ash content, and (4) a higher intrinsic viscosity than the Irrmed cotton.

Each of these factors must be considered from two points of view. (1) Does the difference in question account for the differences in processing performances observed? (2) Does it reflect a genuine difference between raingrown and irrigated cottons rather than a variation between two samples of a similar material? The discussion below will be centered about these two questions.

Length Distribution

Previous studies of fiber length distribution have shown fairly conclusively that this factor has an important bearing on processing performance [17,

TABLE VIII. Properties of Rainmed (R) and Irrmed (I) Fabrics

(Data are for the warp direction only. Differences in the filling direction were comparable to those shown below for all properties tested.)

	48 × 48		80 × 80 Print cloth					
	Grey sheeting		Grey		Dyed		Mercerized and dyed	
	R	I	R	I	R	I	R	I
Grab strength, lb.								
Dry	63	69	51	57	65	63	70	71
Wet	83	82	67	65	77	78	82	85
Grab elongation, %								
Dry	9	10	11	11	7.3	5.7	6.3	6.3
Wet	19	16	22	19	11.3	9.7	10.0	8.3
Ravel strength, lb.								
Dry	62	67	50	52	61	64	66	66
Wet	71	70	55	56	73	71	81	82
Ravel elongation, %								
Dry	9	9	11	10	7.0	6.7	6.0	6.3
Wet	17	15	20	17	11	10	10	10
Wrinkle recovery, deg.								
Dry	—	—	—	—	72	71	75	76
Wet	—	—	—	—	48	50	48	49
Gurley stiffness, mg.	49	55	48	52	36	42	52	36
Gurley impermeability, sec. (0.1 sq. in. orifice)	3.3	3.1	3.8	3.3	9.3	9.8	6.6	7.6
Stoll flex abrasion, hundreds of cycles	17	14	25	21	4.4	5.8	15	17
Shrinkage in laundering, %	10.5	8.6	7.8	8.9	4.3	4.0	2.2	2.4

19]. Cottons with more short fibers and fewer long ones generally process less well than those with more long fibers, although the mean fiber lengths may be very nearly the same. On this basis it would be expected from the facts shown above that the Irrmed cotton should perform better than the Rainmed. Since the reverse was found to be true, it might be concluded that other factors predominated over the effect which might result from the very small length differences encountered.

Fiber Crimp

The influence of cotton fiber crimp or kinkiness on processing behavior has not been definitely established. It is generally agreed that some fiber crimp is necessary in staple fiber yarn manufacture; this is implied by the fact that all successful man-made staple fibers are crimped. Burte [3] has expressed the view that fiber crimp will influence the mechanical properties of wool tops and rovings. Wools with high crimp energy would be expected to make a less compact and, consequently, weaker roving structure. If this is true, then it might also be expected that, other pertinent factors being equal, fiber crimp would also affect all processing steps involving drafting. Spinning would be most critically affected since at this stage the number of fibers in the assembly cross section is a minimum.

In the present comparison the Rainmed cotton, with lower fiber crimp, did in fact exhibit stronger rovings and better spinning performance than the Irrmed. This observation alone does not prove the hypothesis that fiber crimp has an important influence on cotton processing behavior. It does, however, appear to support the theoretical argument that the property and phenomena are related as contributing cause and effect. It suggests further that too much fiber crimp may be undesirable. If, on the other extreme, the absence of fiber crimp precludes satisfactory mechanical processing, then there must be an optimum fiber crimp for maximum processing facility.

Even if it is decided that the Rainmed and Irrmed samples process differently mainly because of differences in fiber crimp, present information will not permit the conclusion that this indicates a basic difference between raingrown and irrigated cottons. Previous study of cotton crimp [16] has shown that samples vary greatly with respect to this property. Not only do fine cottons generally have lower crimp energy than coarse ones, but also among cottons of the same fineness as much as threefold crimp energy differences may be observed. No evidence, direct or reported, has been found to indicate that cotton fiber crimp is related to environmental conditions of growth or to cotton types used in the production of raingrown or irrigated cottons.

Ash and Wax Contents

The data of Table V indicate that the Irrmed sample has more ash and wax than the Rainmed. The difference in ash is believed to be associated with the fact that the Irrmed was mechanically harvested, an operation which may involve somewhat more soil pickup than hand-picking. Information available in the literature does not indicate a basic difference in ash content between raingrown and irrigated cottons [4, 8]. Furthermore, although it is known that ash content affects fiber electrical resistivity [21], and thereby may affect electrostatic charges built up in the fibers, it is not certain that the difference indicated above has caused the difference in processing behavior.

The effects of wax on cotton processing are almost equally obscure. In the present comparison it was observed that Irrmed fibers tended to stick to the roving frame clearers in the 18's yarn manufacture. This occurred only on the very warmest August days. Subsequent observations showed that the Irrmed wax, after extracting and drying, exhibited a lower softening temperature than the Rainmed wax. Accumulations of the wax were also reported on the rolls. These had not been previously cleaned, and therefore no valid comparison could be made with the extracted waxes. Thus, it is possible that the observed differences in wax content and nature may have given rise to the differences in roving performance.

Both ash and wax contents may, of course, affect fiber surface properties such as friction and thereby influence processing behavior. It has been shown that cotton processing is considerably modified by scouring off pectins and waxes [9], by adding lubricants [12], or by adding colloidal silica [2]. The consensus of experienced processors seems to be that scouring greatly increases the difficulties in opening, carding, and drawing operations. Addition of lubricants to cotton is believed to supplement or replace natural waxes needed to make the cotton draw "smoothly." Colloidal silica on cotton seems to re-

duce appreciably the twist multiple required for optimum yarn strength by increasing interfiber friction. In view of these observations, the possible influence of ash and wax contents on the processing differences between Rainmed and Irrmed samples should be investigated further.

Again, however, there is no evidence that the ash and wax contents of these two cottons are representative of raingrown and irrigated cottons. Studies of the quantities of these constituents in various cottons have shown a spread of values which bear no apparent relation to whether the cotton was irrigated or raingrown. Both ash and wax contents seem to depend considerably on the extent of weathering to which the cotton is submitted. Wax contents are closely related to fiber fineness [10, 12] and according to the data of Marsh and co-workers [10] are not otherwise affected by conditions of fiber growth.

Intrinsic Viscosity

The differences in intrinsic viscosity between the Rainmed and Irrmed samples are noteworthy not because they may influence processing but because they confirm a previously reported difference between Deltapine 15 and Acala 4-42 cotton. Intrinsic viscosity values in the ranges observed are not related to measurable mechanical properties of cotton fibers [22]. They may, however, foretell future mechanical performance of fabrics and for this reason are of interest here. Rutherford [13] observed that his samples of Deltapine 15 exhibited a higher intrinsic viscosity than his Acala 4-42. The same was found in the present investigation for the Rainmed and Irrmed samples. If ginned cottons differ in their initial intrinsic viscosity, if these differences persist through to the finished fabric, and if intrinsic viscosity is related to such service characteristics as wet abrasion resistance or laundering failure, then the measurement of raw cotton intrinsic viscosity is a matter of practical importance in cotton utilization. Present evidence indicates affirmative answers to all of these "ifs." The results outlined herein indicate, therefore, that the subject of raw cotton intrinsic viscosity (or molecular weight) as a factor in cotton quality evaluation deserves further attention.

Conclusions

This investigation has failed to uncover any fundamental differences between raingrown and irrigated cottons. The observed dissimilarities between the Rainmed and Irrmed samples were for the most part of minor practical importance. In fact, it seems reasonable to conclude that with careful selection and possibly minor adjustments in processing organization the differences observed in the mechanical processing characteristics of these cottons would become insignificant.

On the other hand, it might also be concluded that the small superiority of the Rainmed sample processing behavior over the Irrmed sample must be related to its lower fiber crimp, or to its different surface properties as these might be affected by its lower ash and wax contents, or to other fiber properties not measured in the present study. Of these the author is inclined to fiber crimp as the dominant factor. However, before this hypothesis can be established as a firm conclusion more study of cotton fiber crimp must be made.

Acknowledgments

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Appendix

Carded Yarn Processing

Processing organization and procedures used in spinning the 18's and 36's carded yarns and in weaving and finishing the fabrics were essentially those described in a previous publication [19]. Four bales of each cotton were used in the 18's comparison and two bales of each for the 36's. The same machinery was used in each with the exception of spinning. Fabrics from both yarns were dyed and finished at the same time.

In the 36's spinning trial [19] differences between cottons and between frames were found. These are illustrated by the average number of end breaks per 1000 spindle hours (for material causes only) shown below.

Frame No.	Rainmed	Irrmed
1	54.81	45.07
2	44.49	58.75
3	40.15	47.37
4	39.77	78.33
5	53.15	53.91
6	56.07	88.74
Mean rate	48.01	62.03

Analysis of variance of the trial in which these results were obtained appeared in Reference [19].

Combed Yarn Processing

Two bales, one of each cotton, were processed together at The American Thread Company plant in Willamantic, Connecticut. Opening and spinning of the cottons were performed in the production areas of the mill, while carding through roving manufacture were carried out in the experimental pilot plant. Plying and mercerizing were also done in the production areas.

The cotton was opened through three Lummis and one Whitin blending feeders, one Spirawhirl Buckley beater, a No. 11 Saco-Lowell condenser and a No. 12 Lattice opener with Buckley beater, and a Centrifair Superior inclined opener and cleaner. Picking was through two Saco-Lowell 2-beater single process pickers. The carding with two H and B revolving flat cards was performed from 12-oz. lap producing at a rate of five pounds per hour 44-grain sliver. One half of each cotton was carded on each of the two cards. Wastes, nep counts, moisture contents, sliver size, and evenness values were taken for each. Breaker drawing was performed on a Medley 4-roll drawing frame with six doublings to produce a 44.5-grain sliver. This was fed into a Model 37 Saco-Lowell lap winder to make three sections for

each cotton so that from these, equal weights of combed sliver would result with 12%, 16%, and 20% noil removal.

A Saco-Lowell comber was used at a speed of 101 nips per minute to obtain a 112-grain combed sliver with the various noil removals. Finisher drawing consisted of two processes with six doublings in each to produce 60-grain sliver. The first was on a 5-roll Saco-Lowell frame, the second on a Medley 4-roll draw frame.

For the roving operation each cotton-noils combination was divided in two for making 2-hank and 4-hank roving, respectively. The 2-hank was made on an H and B Whitin CID roving frame with 4-roll drawing. Twenty-four spindles were used, four for each cotton-noils combination. Halfway through processing spindles for the Rainmed and Irrmed samples were interchanged. A similar procedure was used with the 4-hank roving process which was carried out on a 4-roll H and B Whitin CSD frame with Casablanca system.

Spinning Test

Two Whitin long draft, Casablanca system, spinning frames in the regular production area were used in the spinning test. One was assigned to spin double roving (4-hank), and the other was assigned to spin single roving (2-hank). The Rainmed and Irrmed cottons were spun on opposite sides and reversed halfway through processing. Rovings of different combing levels were grouped separately on the frames.

Spinning was conducted on a 3-shift basis for three successive days and was under constant supervision by laboratory personnel. The same checkers and spinners were used throughout the test. Spindle hours totaled about 12,000 per cotton.

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Modification of Wool with Monofunctional Isocyanates

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Abstract

The reaction of wool with ethyl, octadecyl, phenyl, and α-naphthyl isocyanates under anhydrous conditions is described. At low uptakes these reagents confer marked resistance toward solubilization in hot acid; increasing uptakes reduce the felting shrinkage and impart stability to degradation by alkali and oxidizing and reducing agents.

Introduction

Fraenkel-Conrat, Cooper, and Olcott [4] in this laboratory showed that reaction of several proteins with aromatic isocyanates is extensive in anhydrous pyridine at elevated temperatures. More recently Farnworth [3] noted that similar treatment of wool with phenyl isocyanate increases the stability toward degradation by acids, alkalies, and reducing agents. Since it is highly desirable to confer such stability upon wool, these observations have been extended. In this paper some chemical properties of wool treated with ethyl, octadecyl, phenyl, and α-naphthyl isocyanate are compared. A later paper will consider the effects of modification by several diisocyanates.

[1] Agricultural Research Service, U.S. Department of Agriculture.

Materials and Methods

An 8½-oz. Botany style 404 white wool flannel, thoroughly solvent-extracted and cut into squares weighing 2 g. was used. Isocyanates were commercial products and were used without further purification.[2]

Treatment of Wool

The procedure was, in general, that used by Fraenkel-Conrat *et al.* [4] and by Farnworth [3]. The wool was dried in an oven at 105° C. for several hours and transferred to a round-bottomed flask fitted with condenser and $CaCl_2$ tube. Dry pyridine was added, and when the appropriate temperature was reached, the isocyanate was added. Eighteen milliliters of pyridine and 2 ml. of isocyanate per gram of wool were used throughout the experiments. At the end of the chosen reaction period the wool was quickly rinsed in dry CCl_4. The rinse was immediately followed by extraction for 2 hr. with CCl_4 in a Soxhlet apparatus and finally with ethanol for 8 hr. This procedure avoided troublesome deposits on the surface of the fabric. After washing for several hours in running water, the samples were conditioned at 21° C. and 65% relative humidity approached from the wet side.

Methods of Testing

1. *Acid solubility* was determined by immersing 400 mg. of the flannel in 4*M* HCl for 1 hr. at 65° C., according to the method adopted by Zahn and Würz [9].

2. *Alkali solubility* was determined by immersing the flannel in 0.1*M* NaOH for 1 hr. at 65° C., according to the method of Harris and Smith [5].

3. *Solubility in ammonia after treatment with peracetic acid* was carried out according to the directions of Alexander *et al.* [1]. Four hundred milligrams of wool were treated for 25 hr. with 100 ml. of 2% peracetic acid and finally with 100 ml. of 0.3% ammonia.

Prior to treatment with acid, alkali, or peracetic acid, all samples of flannel were unraveled and cut into small pieces to facilitate penetration of the reagent.

[2] Samples of isocyanates were kindly provided by Mobay Chemical Co., St. Louis 4, Mo., E. I. Du Pont de Nemours and Co., Wilmington, Delaware, and National Aniline Division, Allied Chemical and Dye Corp., Buffalo, N.Y. Mention of specific products does not imply endorsement by the Department of Agriculture over others of a similar nature not mentioned.

4. *The stress at 30% extension* was determined on fibers taken from the flannel. These values were obtained with a constant rate of loading on the apparatus described by Preusser and co-workers [6] and are the results of at least 16 determinations for each treatment.

5. Felting shrinkage was obtained by milling at 1500 r.p.m. for 4.0 min. in the Accelerotor[3] with 0.5% sodium oleate, liquor-wool ratio 50/1. Four-inch flannel squares were first protected from raveling by cementing the edges. The squares were relaxed prior to milling by soaking for 30 min. in distilled water containing 0.05% Triton X-100, a non-ionic detergent, at 40–50° C. Under these conditions untreated flannel shrank by 40% of its original area.

6. Supercontraction was obtained on single fibers exposed to 5% $NaHSO_3$ at 97° C. for 1 hr. [8].

7. Cystine analyses were determined by the Shinohara method after hydrolysis for 5 hr. in 5*M* HCl at 125° C. in a sealed tube [7].

Discussion

The conditions for reaction of the isocyanates with wool, summarized in Table I, vary widely. Whereas considerable uptake of phenyl isocyanate occurs in 0.5 hr. at 97° C., similar uptake of octadecyl isocyanate requires 5.0 hr. of heating. It is possible that the necessity for penetration of the reagents into the fiber under anhydrous conditions is responsible in

TABLE I. Influence of Temperature and Duration of Heating on the Reaction of Isocyanates with Wool

Treatment	Temperature ° C.	Time hr.	Uptake %
α-Naphthyl isocyanate	97	0.5	3.4
	97	1.0	8.2
	Reflux	1.0	39.2
Phenyl isocyanate	97	0.25	1.6
	97	0.50	5.6
	97	0.75	7.4
	97	1.0	12.1
	97	2.0	29.6
Octadecyl isocyanate	97	5.0	3.5
	Reflux	2.0	12.4
	Reflux	7.0	23.0
Ethyl isocyanate	97	0.5	3.7
	97	1.0	10.0
	97	2.0	15.0

[3] Purchased from the Atlas Electric Devices Co., Chicago 13, Ill.

part for the rather severe temperatures necessary. It is also possible that part of the difficulty is due to dimerization of aromatic isocyanates in pyridine. Dimerization is an equilibrium reaction involving dissociation of the relatively inactive dimer to the more reactive monomer at higher temperatures. Aliphatic isocyanates apparently do not dimerize, although trimerization is known [2].

Since treatment of wool with pyridine alone might be expected to alter some of the properties of wool, each experiment with isocyanate in pyridine was accompanied by a treatment of wool without isocyanate. Exposure to pyridine alone affected only the solubility in ammonia following peracetic acid oxidation; this solubility is reduced from 83% for the original cloth to about 55%. The reason for this behavior is not clear, since the cystine contents of the wool were found to be 11.8% before and after pyridine treatment. However, it is possible that a small number of lanthionine cross-links were formed and are sufficient to bring about a greatly reduced solubility. Low uptakes of isocyanates appear to have little additional effect, but the solubility is lowered appreciably below 50% when higher uptakes are applied to the wool. Since the pyridine effect on peracetic acid-ammonia solubility does not appear to increase with time or severity of treatment used in this study and since wools treated with pyridine alone show no other effects, individual control values are not listed. The treatment with pyridine appears to have little effect on the wet tensile properties of the fibers, as illustrated in Table II for stress at 30% extension and Young's modulus.

TABLE II. Stress at 30% Extension and Young's Modulus for Wool before and after Pyridine Treatment *

Treatment	Stress at 30% extension g./gx.	Young's modulus g./gx.
Untreated	0.407 ± 0.023	6.55 ± 0.67
Refluxed in pyridine for 3 hr.	0.377 ± 0.070	6.35 ± 0.69

* Results are the averages of at least 16 determinations and are given in terms of 95% confidence limits.

The most striking result of the treatment of wool with monoisocyanates is the enhanced resistance toward solubilization by hot acid. The data are shown in Figure 1. At the 5% level all reagents except ethyl isocyanate lower the acid solubility of the wool from 9 to less than 3%. Presumably all side chains with reactive hydrogen atoms, namely, amino, hydroxyl, thiol, phenolic, amide, guanidino, and imidazoyl groups, are capable of reacting with isocyanates [3, 4]. The free carboxyl groups also form mixed anhydrides [4]; however, these are known to decompose, either spontaneously or at elevated temperatures [2] and are likely to undergo some change when the wool is dried at 105° C. prior to determining dry weights. The conversion of polar functional groups to less polar side chains may contribute to stabilizing the wool toward acid. Significantly, much larger uptakes of isocyanates are required to give appreciable protection against solubilization in hot alkali.

Solubility in alkali depends in part on the stability of cystine linkages, and Farnworth [3] considers that bulky side chains introduced by treatment with phenyl isocyanate reduce the reactivity of disulfide cross-links through steric hindrance. It is seen from Table III that low uptakes of the higher molecular weight isocyanates afford little protection. However, high uptakes impart appreciable stability toward solubilization in hot NaOH. Wool treated with ethyl isocyanate, however, has a higher alkali solubility than untreated flannel. It is possible that the new bonds formed with this reagent are not stable to hot NaOH. The high alkali solubility is believed not due to derivatives loosely deposited on the fiber because the wool still shows enhanced resistance toward solubilization in hot acid.

The resistance of treated wools to supercontraction should be capable of interpretation in a manner similar to that used to explain increased stability toward solubilization in hot NaOH. The introduction of

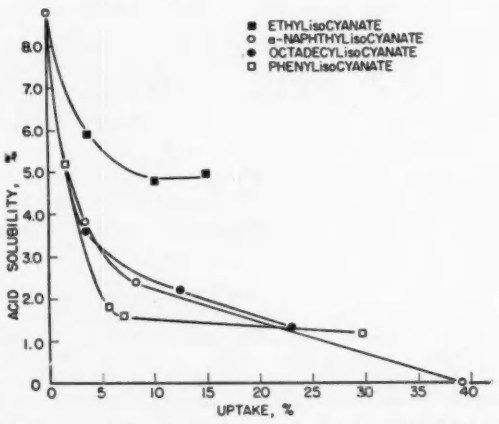

Fig. 1. Acid solubility of isocyanate-treated wools.

TABLE III. Properties of Wools Treated with Isocyanates*

Treatment	Uptake	Acid solubility	Alkali solubility	Peracetic acid-NH_3 solubility	Supercontraction	Shrinkage	Regain
Untreated	0	8.8	12.4	83	27	40	14.4
Pyridine alone, refluxed 3 hr.	−1.0	8.8	12.8	55	27	40	14.0
α-Naphthyl isocyanate	3.4	3.8	11.8	77	14	38	13.6
	39.2	0	7.5	26	3	0	8.5
Phenyl isocyanate	5.6	1.8	12.8	59	15	20	12.6
	29.6	1.2	5.3	16	4	2	7.2
Octadecyl isocyanate	3.5	3.6	12.0	49	23	38	13.5
	23.0	1.3	5.4	19	23	38	8.8
Ethyl isocyanate	3.7	5.9	19.0	65	21	40	12.0
	15.0	5.0	23.5	44	25	39	11.0

* All values, expressed as per cent, are the averages of at least two determinations.

large groups by treatment with isocyanate may be expected to shield the cystine cross-links from attack by boiling bisulfite and to reduce the amount of supercontraction. In support of this idea, treatment with the two aromatic reagents protects the wool structure from supercontraction; on the other hand, neither octadecyl nor ethyl isocyanate had appreciable effect.

The two aromatic isocyanates confer resistance to fabric felting shrinkage, as shown in Table III. At about 6% uptake of phenyl isocyanate the shrinkage is reduced to one-half and at higher uptakes felting is almost entirely eliminated. As in the case of supercontraction, the two aliphatic reagents have no apparent effect on the shrinkage properties of wool.

Appreciably lower values for the moisture regain are obtained for wool treated with isocyanates, as expected for a protein in which polar functional groups are blocked.

At low uptakes of the isocyanates, there is no perceptible change in fabric hand; above 10% of some of the reagents, a boardy feel is noted. The physical properties of isocyanate-treated wools and fabrics have not yet been thoroughly studied, but our preliminary results indicate that wrinkle recovery and fabric stiffness are not significantly altered by moderate uptakes of isocyanates.

Summary

The reaction of wool with ethyl, octadecyl, phenyl, and α-naphthyl isocyanates under anhydrous conditions is described. The reagents confer resistance toward solubilization in hot acid and alkali. The aromatic isocyanates reduce the degree of fiber supercontraction in boiling bisulfite and lower the fabric felting shrinkage, whereas the aliphatic reagents have little effect on these properties.

The solubility of wool in dilute ammonia after peracetic acid oxidation is reduced by heating with pyridine alone but in the absence of the isocyanates other properties including the wet tensile strength are unaffected.

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Cellulose Interactions with Certain Textile Resins

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Abstract

It is known that certain resins when applied to cellulosic fibers will impart wrinkle recovery improvement to the fabric. This improved property is shown to vary directly with the amount of chemical reaction between the resin and the cellulose. The evidence for this is obtained primarily from infrared spectra. These spectra were obtained from cellophane films to which resin had been applied by techniques known to give varying wrinkle recovery. The type of chemical reaction with cellulose is shown to be the same for a number of resins and for formaldehyde.

I. Introduction

A great quantity of cotton and rayon fabric has been treated in recent years with textile resins, such as the melamine-formaldehyde and the urea-formaldehyde types. The use of these resins with cotton and rayon has been largely for the imparting of improved wrinkle recovery properties to cellulosic fabrics which otherwise become wrinkled very quickly when worn. The general method of forming these resinous materials within the cotton and rayon fibers is as follows. The fabric is padded with aqueous solutions of essentially monomeric urea formaldehyde or melamine formaldehyde together with a suitable acid-forming catalyst and other desired additives. After rapid drying, the cloth, now impregnated with the melamine or urea formaldehyde, is heated from a few seconds to a very few minutes at a higher temperature. Curing takes place rapidly in the presence of the catalyst. These types are called thermosetting resins.

An example of the changes in properties of the cloth caused by the treatment of 80×80 cotton with a partially methylated melamine formaldehyde resin [2], Aerotex [1] Resin M-3, is shown in Table I.

[1] Trademark of American Cyanamid Company.

Elasticity and wrinkle recovery values are increased by the resin, as is the ability of the fabric to resist wrinkling when worn and to recover from any wrinkling which may occur. Break load, extensibility at the break, and tear strength are lowered.

The mechanism by which these properties are imparted to cotton by resins of this type has been and still is controversial. The original discovery by Foulds, Marsh, and Wood [3] that resins polymerized within cellulosic fabrics would render the fabrics more crease resistant is now more than 25 years old. The changes in properties, especially elasticity and wrinkle resistance, can be quite conveniently explained by postulating the formation of cross-links [2], and all agents which are really effective for increasing wrinkle recovery are at least difunctional and are theoretically capable of forming cross-links, as stated by Cameron and Morton [1]. The situation is complicated, however, by the number of interactions that are possible. Nuessle, Fineman, and Heiges [4] recently discussed many of the controversial aspects of crease recovery. They list nine possible reactions when a difunctional resin-former (dimethylol urea) is polymerized within cellulose fibers. Reactions 1 to 3 involve only resin poly-

TABLE I. Typical Property Changes by 8% Melamine Resin on Cotton-Filling Direction

	Extension at break, %	Break load, lb.	Wrinkle recovery, %	Elasticity at 10% extension, %	Tear strength, lb.
Control	22.2	31.1	46.1	75.6	0.88
Aerotex resin M-3	16.0	22.3	73.9	89.1	0.64

merization and no bonding to cellulose, but the last six do indicate possible modes of reaction with cellulose as follows: (4) simple cross-link, (5) linear polymer, cross-linked to cellulose, (6) cyclic polymer, cross-linked to cellulose, (7) monomer, linked to only one cellulose chain, (8) linear polymer, linked to only one cellulose chain, and (9) cyclic polymer, linked to only one cellulose chain.

The relative insolubility of resin-treated cellulose in the cuprammonium hydroxide solution used as a means of detecting cross-linking is stressed by Reeves, Drake, McMillan, Jr., and Guthrie [5]. Also it is usually thought that increased moduli and decreased flow, as shown by lowered breaking extensions, are caused by cross-linking.

Cooke, Dusenbury, Kienle, and Lineken [2] have shown by infrared measurements that there is evidence of a chemical reaction between cellulose and resin polymerized within the cellophane.

It is the purpose of this paper to produce further infrared evidence for the nature of the chemical reaction between cellulose and several resins and the dependence of these reactions upon catalysts. All the resin present probably does not react with cellulose as pointed out by Cameron and Morton [1], but this does not affect our conclusions. The data do not completely prove that cross-linking is essential for wrinkle recovery improvement, although it is known in the case of formaldehyde that adjacent glucose units are cross-linked by methylene groups through ether formation. This point is discussed later.

Since it is impossible to obtain infrared transmission spectra of fabric because of its scattering properties or lack of transparency, measurements were made on cellophane, which is a cellulosic material chemically similar to cotton and rayon. Thin cellophane sheets are sufficiently transparent in almost all of the 2 to 15 μ range so that the infrared spectra of resins impregnating the sheets can be studied.

II. Infrared Method

The essential technique was the measurement of the infrared transmission of (1) resin films formed on silver chloride plates and (2) resin deposited within thin sheet cellophane. In the latter case increased absorption was found which was not due to polymerized resin alone or to cellophane. The absorption of resin in the cellophane sheet was measured by a compensation method, that is, unresinated cellophane was placed in the reference beam of a

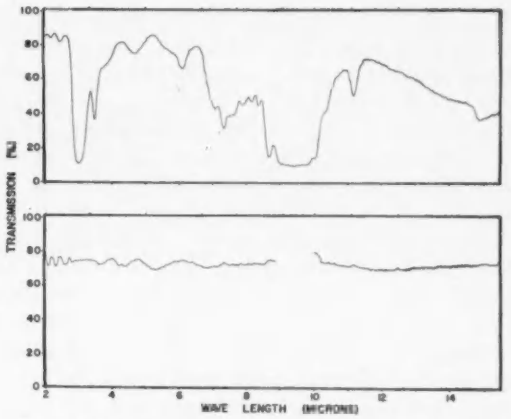


Fig. 1. Cellophane transmission in the infrared and its compensation. Upper curve: uncompensated; lower curve: compensated.

Perkin-Elmer Model 21 double-beam infrared spectrophotometer with the result that only the absorption bands of the resin and any additional absorption caused by new bond formation showed in the resulting curve. The cellophane samples used for compensation had been treated as controls exactly as the resinated sample had been treated except that resin was absent. These cellophane blanks never showed signs of chemical change as indicated by changes in the infrared spectra.

The very thin cellophane sheets were cast by the American Viscose Company. Figure 1 shows compensated and uncompensated infrared spectra of a cellophane sheet which is 7.6 to 8.9 μ thick. The upper or uncompensated curve shows that except in the 3 and 9 to 10 μ region considerable infrared energy is transmitted. The lower curve shows the curve resulting from the compensation technique with cellophane alone in both beams. This curve shows that both beams in the instrument are equal, resulting in the effective cancellation of absorptions. The regularly spaced apparent absorptions are interference patterns whose spacings are determined by the thickness of the film. The space from 9 to 10 μ is left blank in this and in subsequent figures because the cellophane transmission is too little to actuate the recording pen reliably.

III. Resin Reactions with Cellulose

A. Formaldehyde

Evidence for covalently bonded methylene groups was published four years ago by Wagner and Pacsu

[7]. Up to 6% of "fixed" formaldehyde was added through the vapor phase to cotton yarn previously treated with boric acid. They concluded, "the structure of the product of the reaction " (cotton and formaldehyde) "showed that formaldehyde preferentially substitutes the secondary hydroxyl groups of cellulose to yield a structure represented by $R—O—CH_2—O—R'$, where R and R' indicate glucose residues in parallel chains." Previously, there had been considerable study of property changes resulting from reactions of cellulose with formaldehyde under various conditions, and marked changes in properties of the cellulose, such as alkali solubility, swelling, decreased dye uptake, lowered breaking strength, etc., had quite frequently led to the postulate that cross-linking had occurred.

Steele [6] has made some observations of the infrared spectrum of formaldehyde on thin, 2.5 to 5.0 μ, cellulose films. These films were prepared by deacetylating films of cellulose acetate cast from acetone solution on mercury. The films were dipped in a solution of catalyst, usually boric acid, dried, and treated with gaseous formaldehyde at 120° C.

The reactions of cellulose with formaldehyde caused the absorption due to hydroxyls at 2.92 μ to be reduced. Steele concludes that the changes in this case correspond with the expected reduction for the concentration of "fixed" formaldehyde present, assuming that methylene ether cross-links form, thereby reacting a single molecule of formaldehyde with two hydroxyl groups on adjacent chains. Other suggested processes, such as the formation of cellulose hydroxymethyl ether, of hydroxypolyoxymethylene cellulose ethers, or of polyoxymethylene cross-links, would require more than one molecule of formaldehyde per two hydroxyl groups. The formation of unbound formaldehyde polymers would not decrease the hydroxyl absorption.

The additional material added by formaldehyde would be expected to cause the greater absorption found at 3.43 μ, C—H stretching region, due to any of the possible reactions involving cellulose and formaldehyde.

The general increase of absorption found at 8.5 to 11 μ may be due to changes in skeletal vibrations due to the addition of cross-links and/or to the addition of new types of C—O bonds. Similar increases in absorption near 11 μ are found in resin-cellulose studies and are interpreted as due to resin-cellulose bonding.

In the present work on formaldehyde, the thin cellophane sheets were soaked for 5 min. in a solution containing 10% formaldehyde and 1% ammonium chloride. One portion of treated cellophane was air-dried, and another portion was dried and heated at 150° C. for 10 min. A similar treatment on cloth had produced excellent wrinkle recovery and the "fixed" formaldehyde was difficult to remove with acids.

Infrared transmission curves compensated for cellophane are shown in Figure 2 for these materials. The upper curve is for air-dried formaldehyde-treated cellophane. There appears to be no absorption due to formaldehyde or polymerization or reaction products. The curve is very similar to that for compensated cellophane alone. The lower curve is for heated, formaldehyde-treated cellophane. The transmission peak near 3 μ is caused by the decrease in the amount of hydroxyl present. The peak results from overcompensation in the OH stretching region because the actual change is a decrease in the number of hydroxyls in the sample beam. This is identical with the findings of Steele [6]. Decreased hydroxyl absorption can be explained by the loss of some of these groups when they react with formaldehyde to form methylene ethers. New bands at 8.52 and 8.90 μ are normally interpreted at C—O—C absorptions. These new bands would be difficult to observe without the use of cellophane in the reference beam. Although they are not really specific, ether formation probably causes them in this

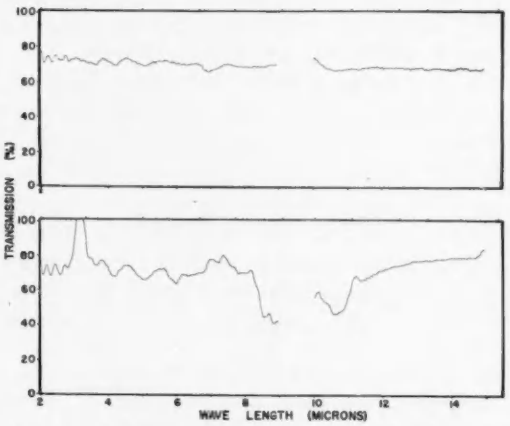


Fig. 2. Infrared transmission of formaldehyde-treated cellophane, compensated. Upper curve for air-dried sample, lower curve for heated sample.

case. The 9 to 10 μ region should not be considered because cellophane absorbs too much energy there. New absorptions in the 10 to 11 μ region could not be assigned to specific functional groups but they must be caused by new chemical bonds. Thus, since the spectrum of treated cellophane is changed markedly by the formaldehyde, there has been chemical interaction between them.

The nature of the reaction of formaldehyde with cellulose is then quite well established, both by the chemical structure proof and by infrared spectroscopy, to be the formation of methylene cross-links.

B. Methylated Melamine Formaldehyde Resins

Evidence for reaction when cellophane impregnated and heated with a partly methylated trimethylol melamine product, Aerotex Resin M-3, and an amine hydrochloride catalyst, Aerotex Accelerator UTX, has been published by Cooke *et al.* [2] and is reviewed below because of its bearing on our present studies. Figure 3 shows the infrared transmission curves form 6 to 15 μ of M-3 films on silver chloride plates. The lower curve is of unheated material containing no catalyst. This same spectrum is obtained when freeze drying techniques are used to remove the water from the monomer solution. The top curve is of a film cured with a catalyst. The 12.28 μ band is characteristic of the melamine ring of the resin. Changes in absorption at 9 to 10 μ are probably caused by changes in C-O bonds but these are difficult to interpret since several different types of ethers may be involved. Another interesting change is the broadening of the 7.40 μ absorption band of the unpolymerized resin and the tendency for this band to shift to 7.50 μ with polymerization. Other infrared curves of uncured M-3 on cellophane show that the acid-forming catalyst causes no change in the spectrum of the resin. Figure 4 shows infrared curves from 10 to 13 μ for Aerotex Resin M-3 uncured and containing no catalyst on silver chloride, on cellophane containing no catalyst, heated, and on cellophane when cured and containing a catalyst.

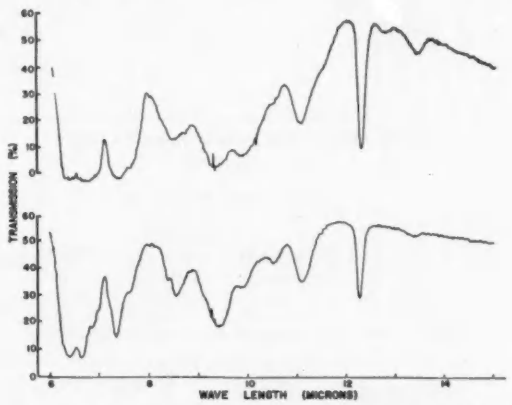


Fig. 3. Infrared transmission of Aerotex Resin M-3 films on silver chloride plates. Upper curve: resin with catalyst, cured; lower curve: resin, uncured, no catalyst.

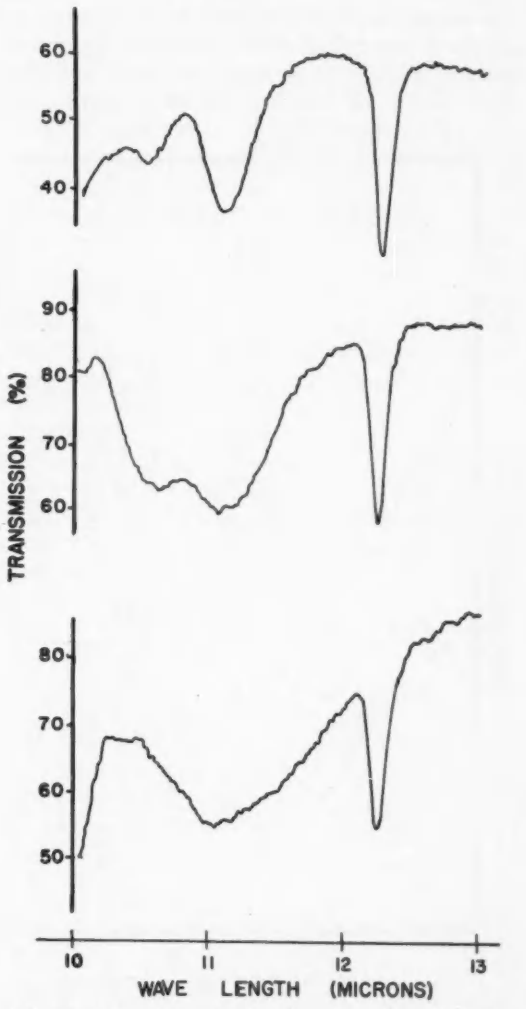


Fig. 4. Infrared spectra showing the curing of Aerotex Resin M-3 in cellophane. Top curve: on silver chloride plate, no catalyst, uncured; middle curve: in cellophane, no catalyst, cured; lower curve: in cellophane, catalyst, cured.

About 16% of monomer solids are present in the cellulose films. Curing of the resin on cellophane produces changes which cause increased absorption near 11 μ. The increase is greater with catalyst present. Similar increased absorption in this region has been shown for formaldehyde on cellophane. Inasmuch as these increased absorptions were shown to be caused neither by curing the resin alone nor by interaction of cellophane or resin with the catalyst, they must be interpreted as being caused by chemical bonds formed between the resin and cellophane.

Catalysts for this resin can be arranged in the order of increasing effectiveness, as judged by changes in wrinkle recovery or other properties. Two metallic chlorides and Aerotex Accelerator MX were chosen which show differing effects. About 16% of monomer solids were added to cellophane without and with one of the catalysts at a concentration equal to 0.0202 mole per 16 g. of resin solids, and infrared transmission curves were made to see if this same correlation would be manifested. Figure 5 shows the infrared curves from 10 to 13 μ of resinated cellophane for no catalyst, and with catalysts, barium chloride, zinc chloride, and Aerotex Accelerator MX, as arranged in order of increasing effectiveness. These curves show an increased absorption above 11 μ which increases with known catalytic efficiency and correlates well with physical property changes. It was previously established that unheated samples on cellophane and heated films of resin on salt plates showed negligible increased absorption in this region. Curing the resin in the presence of cellophane is essential to form reaction bands. The samples had been handled identically as to resin application and heating except for the catalyst present. An absorption at about 10.5 to 11 μ is noted and it seems to be a function of the particular catalyst and to be greatest with the poorest catalyst.

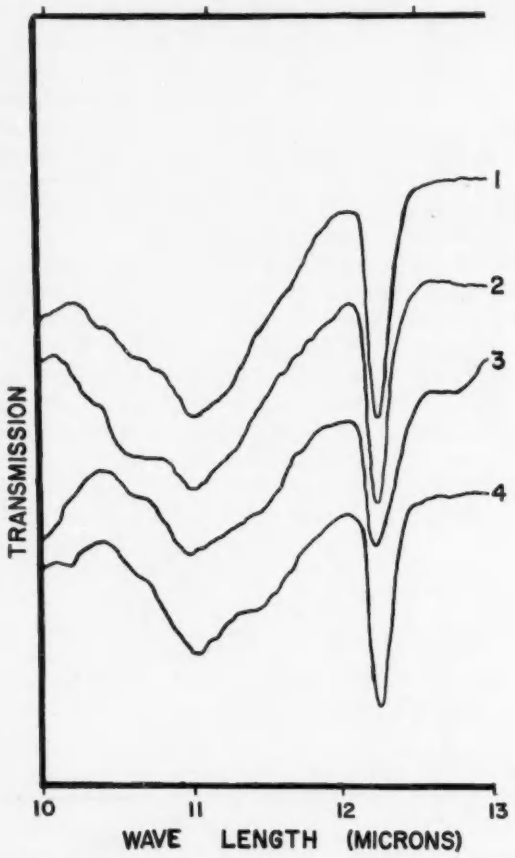


Fig. 5. Infrared spectra showing catalyst effectiveness for curing Aerotex Resin M-3 in cellophane. Curve 1: no catalyst; curve 2: barium chloride; curve 3: zinc chloride; curve 4: Aerotex Accelerator MX.

The effectiveness of catalysts as measured by changes in properties varies directly with the infrared absorption data, as shown in Table II. The applications on 80×80 cotton cloth were 3.5% of monomer solids. This correlation of increased absorption with known catalytic efficiency is very important. This added evidence relates the new chemical bonds causing the increased infrared absorption to wrinkle recovery increase and seems to indicate that the reaction causes the major part of that effect even though the exact infrared assignment at 11 μ is not possible.

TABLE II. Effects of Catalyst on Resin-Treated Fabric Properties

Catalyst	Cloth-resin properties: Wrinkle recovery warp and fill %	Cloth-resin properties: Elasticity at 50% breaking extension (warp) %	Cellophane-resin absorption in 11μ region
None	49	72	Less
Barium chloride	58	78	↓
Zinc chloride	60	82	↓
Aerotex accelerator MX	66	84	More

C. Unmethylated Melamine Formaldehyde Resins

Evidence for interaction of unmethylated methylol melamine with cellulose was also obtained. Cellophane films were treated with about 15% of Aerotex Resin UM, unmethylated methylol melamine, using about 40% of resin solids content of Aerotex Accelerator MX as catalyst. Figure 6 shows the infrared transmission curves for (1) the monomer on cellophane and (2) the cured resin on cellophane. The band appearing in the top curve at 10.2 μ is not present in the curves for resin or for cellophane and is apparently due to resin cellulose interaction. The bonds responsible for this absorption apparently are changed or broken in the curing process since the band is either shifted or removed in the lower curve.

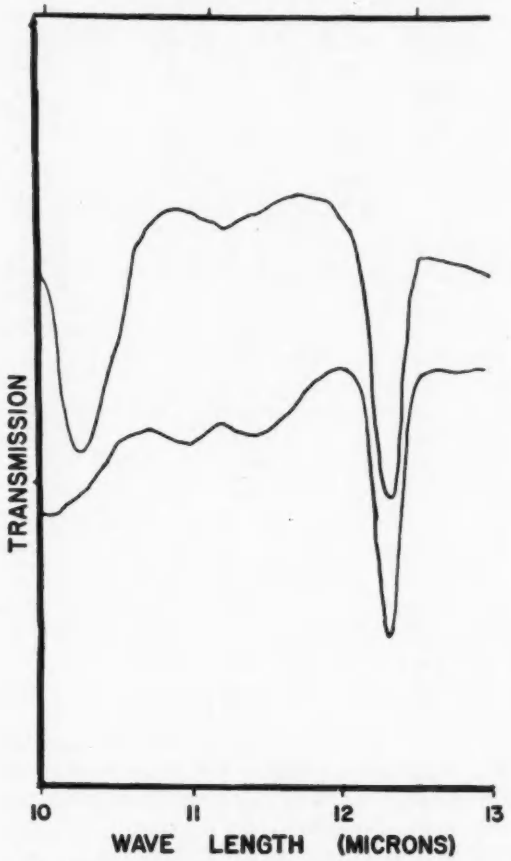


Fig. 6. Infrared transmission of Aerotex Resin UM in cellophane. Upper curve for dried sample, lower curve for cured sample.

The increased absorption at 10.98 and at 11.40 μ present for the cured resin on cellophane is not present in the spectrum of Aerotex Resin UM monomer alone or on cellophane, Aerotex Resin UM polymer alone, or cellulose alone. Since these absorptions appear only in the case of resin cured on cellulose they are probably due to new bonds formed between the methylol melamine and cellulose.

D. Urea Formaldehyde Resins

A methylol urea resin was studied by infrared spectroscopy by the same techniques used with methylol melamine. Applications to cellophane were made from solutions containing 24% of monomer solids of Aerotex Cream 450 which contains both mono and dimethylol urea. When a catalyst was used, it was Aerotex Accelerator UTX (20% of resin solids).

Figure 7 shows infrared transmission curves of films of resin on silver chloride plates. When uncured, there is one absorption in the carbonyl stretching frequency region at 6.00 μ. After polymerization by heating, a second absorption appears at 6.45 μ. This second wave length is usually assigned to N—H vibration of a monosubstituted amide. The changes in this region with curing may be due to substitution in the $—NH_2$ of the monomethylol urea present. The infrared curve of unpolymerized dimethylol urea does show two bands of nearly equal intensity. The intensity of the 6.45 μ band has been correlated with the time of heating. The limiting intensity of the band equals the 6.00 μ intensity. Other changes

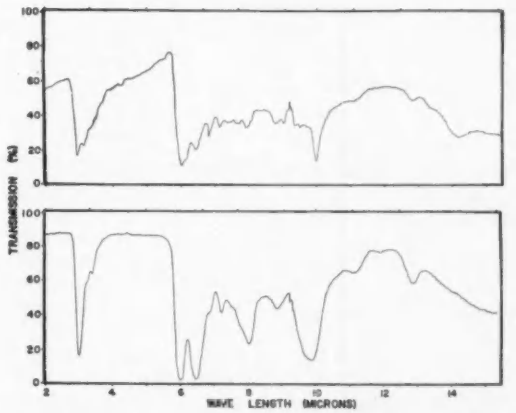
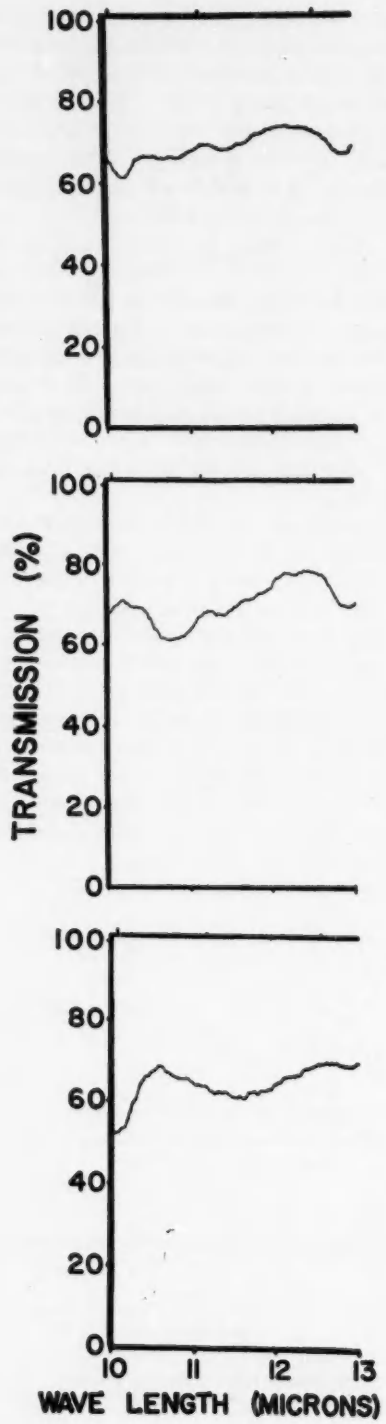


Fig. 7. Infrared transmission of Aerotex Cream 450 on silver chloride plates. Top curve for uncured resin, bottom curve for cured resin.



caused by polymerization are not noteworthy. When the resin is placed in cellophane its spectrum always resembles the polymerized resin. This is indicative of some degree of polymerization or of chemical reaction always occurring.

Figure 8 shows infrared transmission curves in the 10 to 13 μ region for Aerotex Cream 450 on cellophane. The top curve is for uncured resin. The middle curve is for heated resin with no catalyst, and the bottom curve is for cured resin with Aerotex Accelerator UTX for a catalyst. More intense and broadened absorption is found near 11.5 μ for the cured catalyzed resin than for the others. The middle curve shows the beginning of this change. This is not exactly like the interaction band with Aerotex Resin M-3, but it would not be expected to be exactly the same.

Therefore, with a urea-type resin, evidence for reaction with cellulose is obtained which is similar to that found for formaldehyde and for a melamine resin with cellulose.

IV. Acid Stripping

IV. Acid Stripping

Resins are stripped from cotton by the action of a solution containing 5% urea and 1.5% phosphoric acid at 85° C. without much damage to the cloth. The wrinkle recovery and breaking strength values return after stripping nearly to the values before resin application. Research at the American Cyanamid Company has shown, however, that fabric properties, such as wrinkle recovery, change much more rapidly than the resin is removed from the fabric during stripping, if resin removal is judged by nitrogen determinations. In one experiment, one-tenth of the Aerotex Resin M-3 present was removed from cotton initially containing 10% of resin solids after 5 min. in the stripping bath, but 80% of the wrinkle recovery increase due to resin was lost. After 10 min. of stripping, 95% of the increased wrinkle recovery was lost but only 36% of resin was removed.

The loss of improved properties would indicate the acid hydrolysis broke the cross-links before it completely removed the nitrogen-containing material. This is additional evidence for the belief that improved properties are a result of chemical reaction between the textile finish and cellulose.

Fig. 8. Infrared transmission of Aerotex Cream 450 in cellophane. Upper curve for uncured resin; middle curve for heated sample, no catalyst; lowest curve for cured resin, with catalyst.

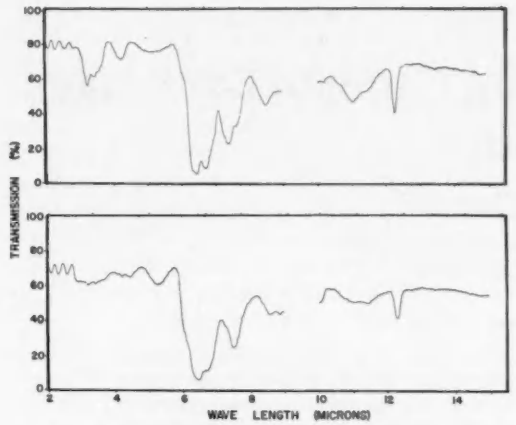


Fig. 9. Effect of brief acid stripping of Aerotex Resin M-3 on the infrared transmission of the resin in cellophane. Upper curve for cured resin, lower curve for partially stripped resin.

Figure 9 shows compensated infrared curves for well-cured Aerotex Resin M-3 with 40% of resin solids of Aerotex Accelerator MX on cellophane and for the same sample after being in a 5% urea plus 1.5% phosphoric acid solution at 85° C. for 5 min. It can be seen that changes have occurred in that brief time: (1) The general absorption near 11 μ has decreased. (2) Resin content has also decreased, as shown by the decrease of the 12.28 μ band. (3) The shifting of absorption from 7.40 to 7.50 μ with broadening indicates the removal of monomer or low molecular weight resin. The lessening in absorption in the 11-μ range indicates that bonds which we attribute to resin-cellulose interaction have, indeed, been partially removed. These changes could account for the large change in properties of treated cotton.

Increased infrared absorption near 11 μ indicates chemical changes which are common for formaldehyde and several resins. In the case of formaldehyde, the evidence, which has been discussed, is strongly in support of methylene groups cross-linking glucose units through ether formation. Infrared absorption which indicates new bonds does not by itself show that the urea formaldehyde and melamine formaldehyde resins form cross-links. Therefore, it can be inferred that the resins cross-link in a manner similar to formaldehyde with the elimination of water or alcohol. The positive correlation of wrinkle recovery increases with increased infrared absorption above 11 μ indicates that the bonding is related to gain in wrinkle recovery. The best indication that cross-linking of cellulose chains must be involved is the rapid loss of wrinkle recovery properties with brief acid stripping without corresponding loss of resin. This loss of properties is far more rapid than is the loss of resin, as judged by loss of nitrogen, *and it is accompanied by lowering of the infrared absorption attributed to chemical bonding of resin to cellulose.* Figure 10 shows the infrared curve for a presumably completely stripped resinated cellophane sample. The absorptions typical of the resin are no longer present.

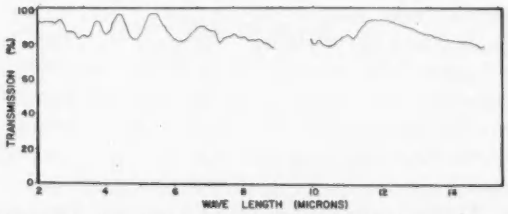


Fig. 10. Infrared transmission of cellophane after completed acid stripping of Aerotex Resin M-3 from cellophane.

V. Summary

Infrared data and their correlation with treated fabric properties have been presented which together with chemical evidence show that chemical reactions take place between cellulose and (1) formaldehyde, (2) melamine formaldehyde resins, and (3) urea formaldehyde resins. These chemical reactions are shown to be involved in the imparting of high wrinkle recovery properties.

VI. Acknowledgments

The authors appreciate and acknowledge the advice, criticism, and help of their associates and especially of Drs. T. F. Cooke, R. H. Kienle, E. S. Pierce, and J. M. Salsbury.

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A Method for Controlling Cationic Exchange in Cotton

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Abstract

The abnormally high copper content of cotton duck which has been impregnated with the solution of a copper salt is due to the adsorption of ionic copper by the cotton. This adsorption may be prevented completely by scouring the fabric and then applying the copper in the form of a water-soluble molecular complex. Citric acid and glycine form such complexes with copper and are effective in preventing its adsorption.

Introduction

In attempting to impregnate 7-oz. cotton duck with small amounts of copper 8-quinolinolate (0.05% Cu), difficulty was experienced in obtaining the calculated add-on. The method consisted in first impregnating the fabric with a known concentration of an acetic acid–water solution of 8-hydroxyquinoline, then immersing the fabric in a bath of excess copper salt to form a precipitate *in situ* of copper 8-quinolinolate. The amount of copper deposited on the fabric by this method was as high as 0.30%, whereas the calculated value was 0.05%. The fabric was thoroughly water-washed before the analysis for copper. When the concentration of 8-hydroxyquinoline was lowered by a factor of 10, the copper left on the fabric after the precipitation step remained the same. From this result it was inferred that the copper was being adsorbed preferentially in the second bath.

The purposes of the work reported here are to (1) determine the cause of this preferential adsorption and (2) devise some means of preventing it.

Causes of Adsorption

A review of the literature on this phenomenon of adsorption by cotton indicated that a considerable amount of pertinent research had been done. Sookne and Harris [7] studied the ash of cotton and other naturally occurring cellulosic materials and concluded that the fiber normally exists as a salt, many of the anions being provided by the cellulose of the fiber and by associated materials such as pectins. Treatment of the fabric with a solution of a salt would result therefore in an exchange of ions, producing a different concentration of metal than would normally be expected.

Walker and Quell [9] showed that cotton which had been water-washed had a higher electrical resistance after washing than before. The washing had reduced the inorganic ash content from 1% of the dry cotton to a value generally less than 0.3%. The water-soluble salts which constitute about 70% of the ash weight were shown by these investigators to be principally sodium and potassium compounds. According to Matthews [3], the ash content of cotton never exceeds 1%.

There is considerable evidence to show that this cation-exchange property of cotton and other cellulosic materials is due primarily to the presence of impurities such as lignin, pectin, and so on. This view is held by McLean and Wooton [4] who demonstrated that the exchange capacity varies markedly with the grade of the fiber. In the course of their work they used acid-washed jute to remove calcium quantitatively from a calcium sulfate solution containing 233 parts calcium per million. The jute eventually lost its exchange capacity, but after regeneration with an acid wash it could be used again to deionize a salt solution. Steuwer [8] showed that pectins are capable of exchange reactions, and this phenomenon is the basis of a British patent granted to Walsh and Adams [10] who state that metal ions, including Ni^{++}, Cu^{++}, and Ba^{++} and similar elements may be recovered with high efficiency from solutions

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of their compounds by passing the solutions through granular insoluble pectic substances.

While the evidence gathered in these studies points overwhelmingly to the role played by pectic substances in the phenomenon of cation exchange, a portion of the exchange process is due to the presence of end groups in the cellulose itself. Sookne and Harris [7] attribute approximately 15% of the exchange capacity of cotton to these end groups. Rabinov and Heyman [5] studied the liberation of acid by cation-free cellulose which was treated with salt solutions. They concluded, as have others, that the acidity of the cellulose is due to groups other than hydroxyl, one of the reasons for this belief being that the alkali and calcium salts formed on interaction of the salts or bases with the cellulose are not readily hydrolyzed.

Certain treatments, such as oxidations in an alkaline medium, increase the exchange capacity greatly. Also cotton cellulose which has been phosphorylated by the urea-phosphoric acid method has an inordinately high exchange capacity. At a phosphorous content of 5%, the cation exchange capacity reaches about 1,000 meq./kg. [2] compared with the value of 66 meq./kg. for depectinized cotton obtained by Sookne and Harris.

Gavoret [1] has shown that anions are not adsorbed to the same extent as cations by cellulose, except at the isoelectric point. One of the results obtained in the current study indicated that the anion was adsorbed to a much less extent than the cation, in agreement with Gavoret's findings.

While the causes of the cationic exchange of cotton have been delineated, there were no recorded attempts found to prevent this phenomenon, except by removing the impurities in the cotton. The problem at hand required the prevention of adsorption of copper without changing the composition or state of the cotton fabric. In the following section the details of several experiments designed to achieve this objective are described.

Experimental

Materials

The fabric used in the initial phase of this study was a 7-oz./sq. yd. cotton duck, unsized. Most of the experiments were performed with this fabric, but in the latter phases of the study a 10-oz. unsized duck was used. Difficulty was experienced in obtaining thorough impregnation with this fabric because of its close weave.

The wetting agent used in these experiments was of the polyoxyethylene-thioether type, and it was usually incorporated into the initial impregnation bath at a concentration of 0.05%.

A. Effect of Ionic Strength Increase

It was felt that a solution rich in monovalent cations might satisfy the exchange capacity of cotton duck so that the simultaneous adsorption of a copper salt would be negligible. To test this hypothesis, two strips of 7-oz. duck were impregnated with different copper chloride solutions, each calculated to give an add-on of 0.05% copper; one solution contained sodium sulfate, the other an equal weight of sodium chloride. The composition of the first solution used was as follows:

$CuCl_2 . 2H_2O$	3.86 g.	
Na_2SO_4	15. g.	Pickup 50%
H_2O	1235. g.	

$$\text{Pickup} = \frac{\text{wt. solution added}}{\text{dry wt. fabric}} \times 100$$

With each solution an add-on of 0.12% copper, instead of the calculated 0.05%, was obtained indicating that this approach was not feasible, at least at these concentrations. In addition, a review of the literature has shown that the divalent cations are more strongly adsorbed than the monovalent, thereby diminishing the effect expected by increasing the ionic strength.

B. Effect of Anion on Copper Adsorption

Strips of 7-oz. duck were impregnated with solutions of copper nitrate, copper acetate, and copper chloride, each treatment calculated to give an add-on of 0.05% copper at 50% pickup. The analyses of the treated fabrics are shown in Table I.

TABLE I

Salt used	% Copper on fabric
Copper nitrate	0.11
Copper acetate	0.06
Copper chloride	0.12

From these results one would infer that the copper acetate would be least subject to adsorption. In later experiments, however, fabrics which had been treated with 8-hydroxyquinoline solution were dipped in a copper acetate bath, and a pronounced adsorption of

copper took place. The reason for this might be found in a study of the various concentrations involved. In the experiment described here the copper acetate was present at a concentration of roughly 4 g./1500 g. solution, while in the later experiments its concentration in the precipitating bath was approximately 25 g./1500 g. solution.

Also, in later experiments in which copper acetate was used in the initial bath, erratic results were obtained, the copper add-on not being consistent.

C. Effect of Complex Formation

Since the ionic character of copper was responsible for its adsorption by cotton, it seemed worth while to apply the copper as an un-ionized molecular complex, such as the one with glycine, shown in Figure 1.

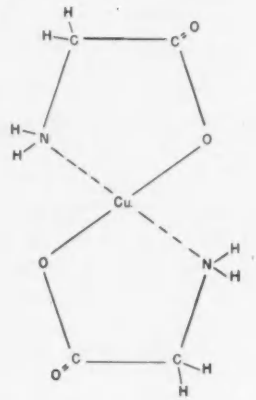

Fig. 1

In this form the copper is devoid of charge, yet the glycine moiety can be readily displaced by 8-hydroxyquinoline to form the insoluble copper 8-quinolinolate. The results recorded in Table II

TABLE II

Complex	Ratio: Mols complexing agent / Mols copper	% Copper on fabric
Glycine	8.36	0.05
Urea	10.60	0.06
Salicylic acid	6.00	0.12
Citric acid	6.56	0.07
Catechol	6.02	0.08
Ammonia	22.48	0.10

show the effects of several complex-forming agents on copper adsorption, including ammonia which of course forms an ionic complex.

Table II summarizes the results of several experiments, each designed for a particular study, hence the lack of uniformity in the ratio of mols complexing agent to mols of copper. Copper acetate was the salt used, the concentration being 3.14 g. in 1000 g. solution. The pickup was 50%.

Glycine and citric acid were the most satisfactory because they controlled the adsorption to some extent and gave uniform impregnations. Urea had an adverse effect on the wetting properties of the solution and was therefore discounted for further study.

D. Effect of Concentration on Complex-Forming Agent

Glycine and citric acid were applied to the 10-oz. fabric at concentrations of 1, 3, and 5 to 1 of copper. The results are shown in Table III.

TABLE III

Complexing agent	Ratio: Mols chelator / Mols copper	% Copper on fabric
Glycine	1.0	0.10
	3.0	0.08
	5.0	0.07
Citric Acid	1.0	0.06
	3.0	0.05
	5.0	0.05

The results indicate that citric acid is the more effective, molwise, in preventing adsorption. A concentration of 3 mols citric acid to 1 of copper prevents the abnormal adsorption of copper since the amount calculated, 0.05% Cu, agreed with the copper add-on. The concentration of copper salt used in this experiment was 2.67 g. $CuCl_2 \cdot 2H_2O$/1,000 g. of solution, and the copper deposited on the fabric in the absence of any chelating agent was 0.10%. At a greater dilution than the concentration used, there is a possibility that adsorption would take place since the complex might be dissociated.

E. Effect of Scouring of Fabric

Sookne and Harris and others cited previously had shown the predominant role played by pectins in the cation properties of cotton. It seemed reasonable to assume that removal of the pectins would diminish the exchange capacity of cotton and thus limit the adsorption of copper. To accomplish the complete extraction of the pectins, 10-oz. cotton duck was

boiled for 1 hr. in 1% NaOH. After washing with clear water, it was dried and then extracted with toluene for several hours in a Soxhlet.

A comparison was made between the copper add-on of this scoured fabric and the regular cotton duck when impregnated with copper chloride, with and without the addition of citric acid as a complexing agent. The citric acid was added at a ratio of 3 mols acid to 1 mol copper in the experiments described here and in the next section. Each of the fabrics was soaked approximately a minute in the copper bath, the calculated add-on being 0.05% copper at 50% pickup.

Table IV shows the results obtained, including the pressure setting of the padder rolls necessary to achieve 50% pickup.

TABLE IV

10-Oz. fabric	% Copper on fabric: No complex	% Copper on fabric: With citric acid	Pressure to give 50% P.U. lb.
Scoured	0.14	0.06	65
Regular	0.12	0.11	25

The results indicate that the scouring process did not prevent adsorption, as was hoped. This may have been due to one or more of the following reasons:

1. The pectins were not completely removed.
2. Oxidation may have taken place during the alkaline boil to create more carboxyl groups which would tend to bind copper ions.
3. By increasing the wettability of the fabric (note difference in pressures needed to give 50% pickup), more naturally occurring carboxyl groups may have have been made available to the solution, thus promoting excess copper add-on.

F. Effect of Soaking Time

The previous experiment had given an indication that the penetration of a scoured fabric by a copper solution was more thorough than that obtained with an unscoured fabric. This naturally gives rise to speculation concerning the effect of the soak time on the penetration and consequent adsorption of a copper solution. Consequently scoured and unscoured fabrics (10 oz./ sq. yd.) were impregnated with solutions of copper chloride, with and without citric acid as a complexing agent, for periods of 2 min., 10 min. and 2½ hr. The pickup in each impregnation was 50 ± 3%. The results are compiled in Table V.

TABLE V

10-Oz. fabric	Solution	Soak time min.	Pressure to give 50% P.U. lb.	Copper add-on
Regular	$CuCl_2$	2	25	0.16%
	$CuCl_2$ Citric acid	2	25	0.08
	$CuCl_2$	10	25	0.25
	$CuCl_2$ Citric acid	10	25	0.12
	$CuCl_2$	150	85	0.28
	$CuCl_2$ Citric acid	150	85	0.11
Scoured	$CuCl_2$	2	75	0.14
	$CuCl_2$ Citric acid	2	75	0.05
	$CuCl_2$ Citric acid	10	85	0.05

For this set of experiments the copper analyses were performed by the carbamate method [6], which is considerably more sensitive than the electrolytic method ordinarily used.

The conclusions to be drawn from this particular study are as follows:

1. Approximately 0.3% copper seems to be the upper limit of the add-on afforded by the solution used (3.14 g. $CuCl_2 \cdot 2H_2O$/1000 g. soln.), at 50% pickup.
2. The add-on after 10 min. is approximately the same as that after 2½ hr., but the difference between a 2-min. and a 10-min. soak is appreciable.
3. Citric acid controls adsorption completely with the scoured fabric but to only a limited extent with the ordinary fabric.

Discussion

The results of this study indicate the complexity of the processes involved in impregnating cotton fabric with a copper salt solution. The adsorption of copper which occurs normally can be controlled, as the results indicate, but some of the factors involved in the absorption process are not clearly defined.

The results obtained with the 7- and 10-oz. duck are not, for example, in complete agreement since the citric acid complex completely controls adsorption by the former, whereas its effect on the 10 oz. is not so great. These two fabrics came from different sources and probably the finishing operations were not the same. This might well account for the difference in results.

Restricting our attention to the results obtained with the 10-oz. duck, it is seen that a scouring treatment must be given the fabric before impregnation with the copper citrate complex in order that cationic exchange be prevented. This process of scouring introduces several known variables which have a bearing on the problem at hand.

1. Removal of pectins: decreases amount of adsorption.

2. Partial oxidation of cellulose: increases amount of adsorption.

3. More thorough wetting: might result in greater adsorption since contact with more end groups could be expected.

The first two factors, removal of pectins and partial oxidation, have opposing effects regarding the process of adsorption. The magnitude of the two might differ appreciably, however, so that it would be fortuitous should they cancel each other exactly. However, some measure of the effect of thorough wetting is possible since the time of soaking was varied. It is not possible to assign a figure to the amount of wetting involved in each case, but some measure is possible since the pressure applied to the rolls in order to obtain 50% pickup was recorded.

Using the pressure as a criterion, it must be said that (1) thorough wetting does not appreciably increase adsorption, and (2) the increase in adsorption by the regular fabric is determined by the rate of displacement reaction rather than by an increase in wetting. These hypotheses are based on the data given in Table IV on the regular 10-oz. fabric. The copper contents of the fabrics treated with copper chloride for 10 and 150 min. were approximately the same, despite the differences in pressure needed to give 50% pickup (25 to 85 lb.). Also the scoured and regular fabrics which were soaked for 2 min. had approximately the same copper content.

No attempt was made to distinguish between the cationic exchange capacity of amorphous and crystalline cellulose.

Acknowledgment

The author wishes to express his appreciation to Samuel Shapiro and Ernest Rohr for impregnating the test fabrics and performing the electrolytic copper analyses. Thanks are due also to Glenn E. Ingraham for the colorimetric copper analyses.

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Manuscript received May 18, 1956.

Letters to the Editor

Short communications in the form of Letters to the Editor are intended to provide prompt publication of significant new research results and to permit an exchange of views on papers previously published in the JOURNAL. These communications are not submitted to formal review as are research papers, and the editors do not assume any share of the author's responsibility for the information given or the opinions expressed. When work previously published in the JOURNAL is the subject of critical comment, the authors of the original paper are given an opportunity to submit a reply, which will be published concurrently when possible.

Static Electrification of Filaments

TEXTILE RESEARCH INSTITUTE
Princeton, New Jersey
September 11, 1956

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In continuing the work on the static electrification of filaments, using the apparatus described by Hersh and Montgomery [1], certain interesting results have been obtained.

Work by Hersh and Montgomery [1] had shown that for insulators rubbed on insulators, the charge generated appeared to be independent of the velocity of rubbing, except where Teflon was involved. In continuous rubbing experiments, however, grounding but not discharging the filaments before the start of each stroke, we have found that velocity has a considerable effect on the quantity of charge developed with most of the insulators tested. For example, increasing the velocity from 2.52 cm./sec. (5 rubs per minute) to 7.65 cm./sec. (15 rubs per minute), doubled the charge for nylon on acetate and acetate on nylon, and increased the charge by 50% for acetate on polyethylene and polyethylene on acetate.

In some experiments with polymer blend fibers (polyacrylonitrile–cellulose acetate in different proportions), unusual effects were noticed, the sign of the charge produced being found to change after a certain number of rubs. Extended experiments with continuous rubbing, both with polymer blend fibers and with cellulose acetate rubbed on nylon, suggest that there is a directional effect related to the drawing of the fibers, which governs the sign and perhaps the quantity of charge developed. It is interesting to note that such reversals of sign have been observed in practical setups where assemblies of fibers are run continuously and the static charge developed is measured.

Experiments on the discharging of a nylon filament charged by rubbing have been performed, using the radiations of polonium and radium sources. The polonium source was prepared by plating from a hydrochloric acid solution the contents of very old radon capsules. Thus, neglecting the beta radiation of a slight amount of RaE which might have been present and the weak gamma radiation of the polonium, this source was essentially a pure alpha particle emitter. The radium source contained radium in equilibrium with its decay products and thus emitted appreciable beta and gamma radiations as well as alpha particles. It was observed that complete discharge took place when the filament was exposed to the radium source, but when the polonium source was used, an appreciable portion of the initial charge was not removed. This leads us to conclude that, in certain cases, part of the triboelectrically generated charge resides within the volume of the fiber and thus can be removed only by exposure to penetrating beta and gamma radiations.

JOSEPH B. LEVY
J. H. DILLON

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The Bilateral Cortical Structure of Pakistani Carpet Wool

Textile College
Gordon Institute of Technology
Geelong, Australia
August 29, 1956

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

Evidence is presented that a bilateral cortical structure is shown by many nonmedullated fibers from Pakistani carpet wools in fiber thickness up to 35 μ. Medullated fibers typically showed an ortho-cortical annulus around the central medulla. The tendency to a central ortho-cortex increased in non-medullated fibers with increase of fiber thickness.

Introduction

Dusenbury and Jeffries, as reported by Dusenbury and Menkart [2], in a characterization of B.A. fleece wool of mean diameter of the order of 50 μ, noted that there was no indication of structural asymmetry and suggested that the two cortical components may be present, but randomly distributed.

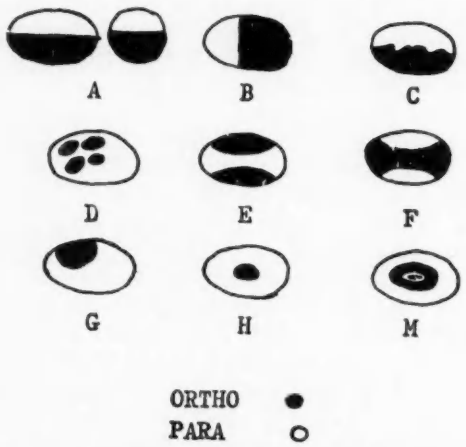

Fig. 1. An arbitrary classification of cortical segmentations types (basophilic staining); *a*, clear demarcation along major axis of ellipse (also includes circles); *b*, clear demarcation along minor axis of ellipse; *c*, gradation from ortho to para; *d*, ortho in several groups; *e*, central channel of para; *f*, *e* in reverse; *g*, peripheral small occurrence of ortho; *h*, central ortho, peripheral para; *m*, medullated fiber, with ortho surrounding the central medulla.

The opportunity having occurred for examining the cortical basophilic staining of 14 Pakistani carpet wools, with Methylene Blue, and the results having shown a definite cortical segmentation, sufficient interest was displayed for a report in detail.

Procedure

After degreasing in several rinses of light petroleum (B.P. range 40°–79° C.) at 15° C., and washing in distilled water, the samples were dehydrated with alcohol and dried. Dyeing was performed with Methylene Blue 0.12% by weight in 0.03 *M* potassium dihydrogen phosphate buffer (pH of 7.4) at 100° C. for 30 min. Cross sections were examined in Euparal mountant.

Microprojections at 500 diameters were outlined on millimeter graph paper so that estimations of area would be available. Para-cortex (H cells) and Ortho-cortex (S cells) were discriminated by the differential staining, in the ideal case the para-cortex being unstained.

Results

It was noticeable that cortical segmentation was unmistakably present and that there were types of segmentation present as shown in Figure 1 (using the nomenclature of Ahmad and Lang [1]).

Types D and E did not occur in these samples, and in any case they are comparatively rare, but were included here for completeness of nomenclature.

Percentage of Para-Cortex

In Table I, where the percentage of para-cortex and the mean thicknesses of nonmedullated and medullated fiber groups in each sample are given, the limitation of numbers must be recognized, particularly in respect to the medullated fibers. The area of para-cortex compared with the area of the fiber (including medulla in the fiber area in the case of medullated fibers) does not appear to be greater in the coarser medullated group, than in the finer non-medullated fibers. In both cases, para- and ortho-cortex are approximately equally distributed.

Types of Cortical Segmentation and Their Mean Thicknesses

In Table II, the numerical occurrence of the types of cortical segmentation in nonmedullated fibers is

TABLE I. Percentage by Area of Para-Cortex in Medullated and Nonmedullated Fibers

	Nonmedullated fibers			Medullated fibers			
	Mean thickness (μ)	% Para-cortex	No.	Mean thickness (μ)	% Para-cortex	% Medulla	No. of fibers
Kaghani	29	46	40	43	62	15	9
Harnai	21	43	49	30	67	9	2
Punjab	25	52	35	32	46	19	14
Bibrik	24	59	42	41	49	10	6
Thall	24	55	37	34	51	16	7
Montgomery	23	53	41	40	57	8	6
Khorasan	21	46	36	35	59	7	13
Sindhi	26	51	30	40	49	18	18
Pahari	24	47	44	42	47	19	5
Peshawre	27	50	46	55	59	22	4
Khandhar	24	55	44	45	44	10	4
Bahawalpuri							
No. 1	21	51	39	36	54	15	11
No. 2	22	41	45	37	48	11	5
Punjab (Superior White)	26	47	40	39	58	14	9
		49.7			53.6	13.8	

TABLE II. Types of Segmentation and Their Mean Thicknesses in Nonmedullated Fibers

	Type of segmentation Number						Mean fiber thickness of each group (μ)					
	A	B	C	F	G	H	A	B	C	F	G	H
Kaghani	15	1	15	1	—	8	26	22	29	36	—	32
Harnai	19	5	13	2	1	9	18	15	23	27	27	28
Punjab	3	—	16	—	7	9	22	—	24	—	24	28
Bibrik	8	1	4	—	16	13	20	16	19	—	25	26
Thall	7	—	20	—	4	6	22	—	23	—	22	28
Montgomery	7	2	14	—	10	8	21	24	23	—	23	26
Khurasan	17	3	11	—	4	1	18	20	23	—	25	27
Sindhi	12	—	3	2	10	3	23	—	25	29	30	29
Pahari	16	1	6	—	6	15	21	28	21	—	25	29
Peshawre	17	1	16	—	10	2	25	22	29	—	28	32
Khandhar	16	—	5	1	5	17	21	—	21	40	23	26
Bahawalpuri												
No. 1	23	4	13	—	5	—	22	20	24	—	21	—
No. 2	24	2	6	—	6	1	20	17	21	—	27	24
Punjab (Superior White)	18	—	12	—	6	4	25	—	26	—	25	33
Totals	202	20	154	6	90	96						

given, together with the mean thicknesses of each group. Since all medullated fibers were of type M with a central medulla surrounded by ortho-cortex, their numbers and mean thicknesses can be obtained from columns 4 and 7 of Table I, and there is no need to tabulate them separately.

Type H (central ortho-) is always of greater mean thickness in any nonmedullated sample than Type A, and in general, with two exceptions, so is Type G. Type F which is infrequent, tends to occur in fibers at the higher limit of range of thickness. Type A in general, has a finer mean than Type C, and this is of the order of 1–5 μ. In two instances there is no difference and in another, the reverse is the case. It is usual for Type B to be finer than Type A, but exceptions occur. In Table III, the distribution in types is subdivided into the following arbitrary diameter classes: 25 μ and under, 26–35 μ, and 36 μ and over. These give an opportunity for comparison with Fraser and Rogers [3] observa-

TABLE III. Division into Segmentation Types on an Arbitrary Diameter Basis

	Fiber segmentation types																		
	25μ and under						26μ–35μ							36μ and over					
	A	B	C	F	G	H	A	B	C	F	G	H	M	A	B	C	F	G	H	M
Kaghani	7	1	2	1	—	—	8	—	13	—	—	8	1	—	—	—	—	—	—	8
Harnai	19	5	10	1	—	3	—	—	3	1	1	6	2	—	—	—	—	—	—	—
Punjab	3	—	11	—	5	2	—	—	5	—	2	7	12	—	—	—	—	—	—	2
Bibrik	8	1	4	—	9	7	—	—	—	—	7	6	2	—	—	—	—	—	—	4
Thall	6	—	18	—	4	2	1	—	2	—	—	4	4	—	—	—	—	—	—	3
Montgomery	5	2	11	—	7	4	2	—	3	—	3	4	2	—	—	—	—	—	—	4
Khurasan	16	2	8	—	2	—	1	1	3	—	2	1	6	—	—	—	—	—	—	7
Sindhi	5	—	2	—	1	—	7	—	1	2	8	3	7	—	—	—	—	—	—	11
Pahari	15	—	6	—	3	4	1	1	—	—	3	8	—	—	—	—	—	—	3	5
Peshawre	10	1	5	—	3	—	7	—	10	—	5	2	—	—	—	1	—	2	—	5
Khandhar	15	—	5	—	4	9	1	—	—	—	1	6	—	—	—	—	1	—	2	4
Bahawalpuri																				
No. 1	17	4	10	—	4	—	6	—	3	—	1	—	3	—	—	—	—	—	—	2
No. 2	24	2	6	—	2	1	—	—	—	—	4	—	6	—	—	—	—	—	—	5
Punjab	9	—	6	—	3	2	9	—	6	—	3	—	2	—	—	—	—	—	2	6
Totals	159	18	104	2	47	34	43	2	49	3	40	55	47			1	1	2	7	66

tions, which are that for wool of less than 25 μ diameter, the segments are discrete; between 25 μ and 35 μ the demarcation is less sharp and the uptake of dye variable, with ortho-cortex surrounded by para-cortex and vice versa; and above 35 μ the division between ortho- and para-cortex is no longer justifiable, the ortho- tending to be at the center, in the coarser fibers.

Reference to Table III shows that fibers with marked segmentation (Types A and B) are common at higher diameters than 25 μ, although it would be approximately true to claim that the mean thickness is of the order of 25 μ. Types A and B did not occur over 35 μ. Conversely Types G and H (central ortho-) were nearly as prevalent in the 25 μ and under group as in the 26–35 μ. Type C, which could have been expected to occur more in the over 25 μ groups actually did so more frequently in the 25 μ and under groups.

Discussion

The intense staining of medulla and the surrounding ortho-cortex in medullated fibers suggests a possible close association in their origins in the follicle papilla. The increasing tendency to Type H as the nonmedullated fibers become coarser may be relevant to this supposition.

The finer fibers in these Pakistani carpet wools show marked bilateral segmentation, as well as the types of section where the edge of the demarcation is less clear and also where the ortho-cortex is located centrally. This is in contradistinction to the normal growth in Merino fine wools which show only the first of these.

Acknowledgments

Our thanks are due to the Director of the Wool Testing House, Karachi, for supplying the samples and to the Colombo Plan Administration through whose generosity one of us (N.A.) has been enabled to make this study.

NOOR AHMAD
W. R. LANG

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INDUSTRIAL SECTION

TRI Annual Meeting

The following five papers were presented at the Annual Meeting, March 22 and 23, 1956.

Are We Thinking of the Washer and Wearer?[1]

Jules Labarthe[2]

Mellon Institute, Pittsburgh, Pa.

THE washer and wearer is either the one-time customer or the repeat purchasing consumer of our textile products. Which role she takes depends entirely upon the extent to which the performance characteristics and the care requirements, respectively, have satisfied her needs and fitted in with her customary methods of washing and caring for her textile purchases. Regardless of whether we use the term "wash and wear" (a most misleading description) or the preferred "minimum care fabric" description, these products definitely fall into the category of "do-it-yourself items" for the homemaker. Thus by definition as well as intention, their promotion is based upon ease of washing and drying, the minimum amount of ironing, the maximum in crease retention and wrinkle recoverability, and, sometimes, spot and stain resistance.

Conditions Vary

One or more of these attributes will attract store customers to the merchandise, but there are vast differences among washing conditions, appliances, and washing materials. Also, two homes with the same number of children may differ as much, one from the other, in wear and care of textiles, as would a couple using their apartment house laundry differ from the career girl who washes things out by hand more frequently than she resorts to a washing machine. Minimum care fabrics are promoted as being all things to all people. Each of us, I think, has been guilty of emphasizing so greatly the ease of washing, drying, and minimum pressing that we have led the consumer to believe that these readily hand-washable, drip-dry, and ironing-free fabrics must always be hand washed and are incapable of withstanding home laundering equipment. This type of emphasis seems to lead the advertising copywriter including, I must admit, the copywriter and artist for store advertising departments, to describe and to picture the washing of these textile products as a finger-tip operation. Soil and stains must be removed from these garments, and the consumer will soon find that they do not fall into a new super-ease of washability classification such as "finger-tip washable" or "dainty dunkable." The manufacturer's label sometimes advocates an utterly unrealistic wash condition. Not long ago our laboratory rejected for sale a glazed cotton bedspread that was promoted partly on the basis of its wrinkle resistance and "that it required no ironing." The washing direction tag stated that this bedspread should be washed by hand in a stationary tub or in the bathtub, then hung up to drip-dry. The ads never depict the puddles of water on the floor under drip-drying articles. It was our belief that if the finish and dyestuff on this particular bedspread required that kind of washing operation, then it should not be sold in the store.

[1] Presented at the 26th Annual Meeting of Textile Research Institute, Symposium on Minimum Care Fabrics, March 22, 1956.

[2] Administrative Fellow.

Is Special Washing Necessary?

Much of our emphasis has been on the convenience of this kind of material in the clothing of the man who travels or the girl who wants to retain that all-day freshness in her clothing so that she will still appear well-groomed in the evening if an unexpected date develops during the day. Less emphasis has been placed upon the serviceability of these fabrics in clothing for the children, or for the housewife herself, or for leisure clothing or sportswear. Surely these minimum care fabrics have a real place in the wardrobes of all elements of our economy and within all age groups. This widespread acceptance, however, will not develop unless they wear satisfactorily, and unless their washing and care do not *require* extra steps, extra procedures, and different drying conditions than those customarily used by that family making the purchase. Unless it is over the shower stall or bathtub, the usual apartment house dweller, whether family or career woman, has no place to hang articles for drip-drying after washing. No housewife objects to some hand washing of clothing, but for such potential customers and consumers, these products must be capable of going into the usual wash load and possibly even the household dryer if the apartment house has such equipment. Indeed in many of our American homes, the household dryer is proving itself to be one of the most convenient of all labor-saving devices, but do all articles that retain their creases when drip-dried retain them when they are dried in a household dryer?

Fiber Identification

Home economics teachers, agricultural extension workers, editors of the women's magazines, and other advisors to the consumer are aware that this new convenience of minimum care fabrics is a desirable characteristic of the synthesized fibers that is now being achieved, too, by the natural fibers through chemical processing. The grouping of all minimum care fabrics with nearly identical performance claims is, I believe, very likely to cause a great deal of perplexity, for the over-all wash and wear or minimum care motif is likely to carry more weight in the mind of the consumer than the type of fiber used in the fabric unless it bears a sewn-in label identifying the fibers of which it is composed. I am thinking now of the housewife whose first minimum care fabric purchase is a crease-resistant rayon or cotton. She may find that although the label calls for drip-drying, it can go into her automatic dryer and then, if she wishes, she can touch it up a bit with a hot iron much more easily than ironing untreated materials in this same wash load. Her next purchase of minimum care textiles may be in a Dacron and rayon blend, or some other synthesized fiber. The claims being the same and the fiber identity not being of much significance to her because it was not stated on a permanent label, this purchase may be treated in the same manner and physical damage to the fibers will result. On the other hand, the first purchase may delight her because of the ease of drying of the all-synthesized fiber article, whereas the next fabric containing hydrophilic cellulose fibers may dry much more slowly and with larger puddles on the floor and make her doubt the value of this kind of merchandise.

There is a conscientious desire on the part of most of today's producers of these minimum care fabrics to develop a uniform and reproducible test method for their evaluation and truly descriptive terminology for labeling the fabric performance. Representatives of many of these textile firms are cooperating with the minimum care fabric (or wash and wear fabric) test method task committee of the American Association of Textile Chemists and Colorists. The interlaboratory testing program is soon to begin. A considerable yardage of materials has been made available to this committee. It is essential, I think, that the fiber identity be disclosed on permanent labels. This is a controversial matter, nevertheless, this information does give the consumer a clue as to the kind of care she should give the article. I well remember a minimum-care dress purchased for a particular meeting and dinner in New York. When this woman unpacked her suitcase, she found the dress slightly mussed and thought the valet in the hotel might touch up the hem a bit with an iron, for she believed the time too short for the dress to hang out before she had to wear it. The fiber identity was not disclosed on the label, and it happened that the fabric was a blend of Dynel and wool. When the valet pressed it under his steam pressing machine, the result was complete catastrophe, for the Dynel melted. This is not intended to be a condemnation of Dynel or any other fiber, but had the customer or the presser known the type of fiber, the disaster could have been averted.

Incidentally, we recommended that the customer be given full credit for this dress.

I would also like to illustrate the rather queer thinking of some textile producers on the subject of fiber identification. Not long ago in a conversation with a producer of fine fabrics designed, I believe, for the high fashion market, the statement was made that identifying the fiber in a blend would mislead the consumer. It was a rather surprising observation, but then this producer went on to say, "There is a certain blend I make that contains acetate as one of the fiber constituents. Now if that fabric were to be labeled "acetate," consumers would believe that it was a washable material because they know that acetates are washable. I know this fabric cannot be washed. As a matter of fact, it cannot be dry-cleaned either. It water spots, is subject to perspiration damage; the filling yarns are very slippery, and if the dress fits too tightly or if the wearer makes a sudden movement, the seams will open up. In fact, the best thing that the wearer can do when the dress becomes really soiled is to discard it." Perhaps I looked somewhat startled; I know I was shocked, and then he went on to say, "We have made hundreds of thousands of yards of this material, and we've had no customer complaints. I was a little surprised that we've had none, but that is the truth." A few days before Christmas, I saw three dresses of this type of material that were purchased in June and were returned by customers because "they could not be cleaned" or "the seams opened." None of you would like to purchase an item made of a fabric which the manufacturer himself knows is not serviceable but which, being a high-style item, rarely if ever reaches a store laboratory for appraisal except in the form of a consumer complaint? In this case, I would like to know who is cheating whom, and yet I will charge that any manufacturer who knowingly produces a fabric of such poor serviceability is discrediting the fiber of which the fabric is made; and to label such fabrics as to their fiber identity would do a disservice to that fiber or to that fiber combination. The point I want to stress is that no fiber, no fabric, no type of promotion is entirely safe from the mistreatment of unthinking, careless, or chiseling producers.

Many Sales Appeals Available

To make consumers out of customers means a constant selling job, not only of the basic idea of these minimum care fabrics but also of the individual advantages of certain fibers and certain blends. All of these mixtures surely will not appeal to the same type of consumer use and experience. To some extent, the textile product itself will determine whether it is going to be worn by a householder or an apartment dweller, by a housewife or by a career girl in the office. Certain basic questions must, therefore, be answered by the producer, by the fabric weaver, or by the finisher if the retail-store buyer is to stay sold on this kind of merchandise, and if consumers are to continue to welcome it, not just as a gimmick, but as a permanent part of their wardrobe requirements.

1. Is this fabric or the article made from it truly washable?

2. Must it be washed by hand, or can it withstand moderate or even hot (130°–140° F.) water in the home washing machine?

3. Can it be washed with a full wash load without having wrinkles formed in it?

4. Is a special detergent required in order to cleanse these fabrics and to help retain the crease?

5. Can the fabric withstand a spin dry operation without loss of the crease and without the sealing in of wrinkles?

6. Will the fiber withstand the conditions in a typical household clothes dryer?

7. Will hot tumbling affect the appearance of the fabric or the article made from it in terms of wrinkling and mussing?

8. If drip-drying or some other procedure is essential for the preservation of the crease in the article or for the safety of the fiber, then are you prepared to have that information on a permanent sewn-in label on the garment?

9. How receptive will you, as a textile producer, be to complaints and comments about unfortunate experiences, and will such justified complaints be regarded by you as a mandate to make a change in fiber, fabric, or finish to help make a consumer out of this customer?

Minimum Care Cotton Fabrics[1]

Paul B. Stam,[2] George S. Y. Poon, M. Joanne Spangler, and Marks P. Underwood

Dan River Mills, Inc., Danville, Virginia

THE production, evaluation, and consumer acceptance of cotton fabrics with "minimum care" properties involve the interaction of many complex factors—technological, economic, social, and psychological. Even the moral factor of promotional claim validity is an important consideration.

Cotton fabrics with minimum care properties are not entirely new. The fact is that for years many cotton fabrics have been relatively easy to recondition after use. The majority of cotton fabrics can be washed with a minimum of special attention using readily available home laundry equipment or relatively inexpensive laundry service. This aspect of ease of care required a long and concerted effort to attain. The coordinated efforts of the chemical industry to provide suitable dyestuffs of several different classes and of the dyeing industry to develop proper methods of application have changed the level of colorfastness of cotton fabrics during the last twenty years. During the same period the cotton industry attacked the problem of fabric shrinkage by better utilization of fabric geometry and construction and by the widespread use of compressive shrinkage equipment. The consumer now expects dyed cotton fabrics to be unconditionally washable under severe conditions of washing temperature, washing chemicals including bleach, and vigorous mechanical agitation even under the conditions of tumble-drying which have recently become more widely used.

The amount of literature on the subject of minimum care cotton fabrics is small. Lippert [3] pointed out the large range of fabric and processing variables which have a bearing on performance. Coloration and fabric geometry are particularly important. The fact is, however, that in most cases the finisher whose objective is to improve minimum care properties is not able to significantly modify the basic fabric geometry or the coloration pattern in order to accomplish this. These factors have traditionally been predetermined by merchandising consideration. Even when working under optimum conditions of fabric geometry and coloration, a cotton fabric must be chemically modified in order to meet today's consumer expectation for minimum care cottons. In a recent paper, Williams [4] presented a strong case for the direct relationship between crease recovery and wash and wear performance as evaluated by a panel which judged the amount of pressing required. This direct relationship exists only under the same conditions of coloration and fabric construction and emphasizes that recent developments in crease-resistant finishes are improvements in degree rather than in kind.

Crease recovery in this paper has been measured by the TBL method, as outlined by Tootal, Broadhurst, Lee Co., Ltd., in "Memorandum of Tests for Cotton, Rayon, and Linen Fabrics," July 1945. Since the Monsanto method, as outlined in ASTM D1295-53T, is also used in the industry, Figure 1 illustrates the values obtained by the two methods on a premium grade 80 × 80 print cloth used throughout this work. Two resins, a methylol melamine (MF) and dimethylol ethylene urea (DMEU), were applied with two separate catalysts: 2-methyl-2-amino-propanol-1 hydrochloride (AMP), and magnesium chloride. Each point represents the mean of 5 warp and 5 filling crease recovery measure-

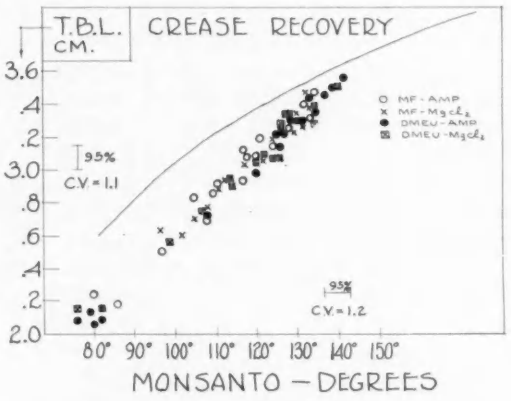

Fig. 1

[1] Presented at the Annual Meeting, Textile Research Institute, March 1956.

[2] Present address: Burlington Industries, Inc.

ments by each method for each of two samples to which resin and catalyst had been applied on two different days and tested by two different operators. In addition, a sufficient number of duplicate samples were tested by the same operator in order to allow for a statistical analysis of operator variables. The 95% confidence range of the averages is indicated by the symbols near the scale and the coefficient of variation of the mean 1.1 and 1.2 indicates a slight advantage for the TBL method. Actually both methods gave good statistical data. The solid curve indicates the calculated relationship between the length of the chord and the corresponding angle of recovery of a 4-cm. sample. It is evident that the TBL method gives less optimistic values for measured crease recovery.

If such an excellent relation between the two methods were universally found, it would be relatively easy for the industry to settle on a single testing method. Unfortunately this relation is not found when unbalanced fabrics are tested or when the formulation is complicated by the addition of thermoplastic resins or softeners. Even for an 80 × 80 cotton, an interesting difference between the two methods is observed. Figure 2 shows the frequency distribution for the difference between warp and filling recovery for about 300 samples. The dashed line represents isotropic behavior. By TBL there is obviously a strong tendency toward anisotropic results, the warp showing higher recovery than the filling. The Monsanto method exhibits isotropic behavior. Actually the anisotropy exhibited by TBL measurements is not unexpected in view of the difference in twist between warp and filling and the very different tension and relaxation history in the two directions in mercerized fabrics. So for these and other compelling reasons this laboratory intends to use TBL measurements until adequate explanations of these anomalies emerge.

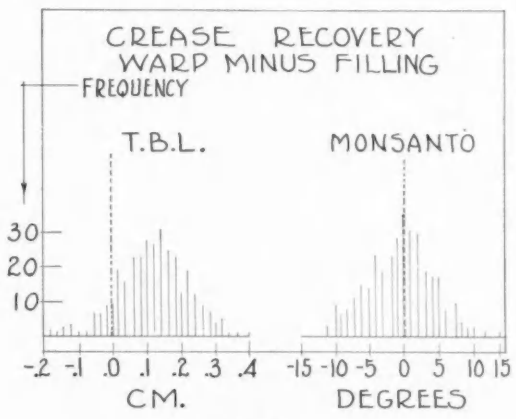

Fig. 2

Conventional crease-resistant finishes of high quality produced during the last few years have had crease recovery by the TBL method in the 2.9 to 3.0 cm. range, approximately 110° to 120° by the Monsanto test method. The requirements for a good wash and wear performance on a solid shade, plain weave cotton fabric are a minimum of 3.3 cm., preferably 3.4 or 3.5, corresponding to 130° to 140°.

The following experimental data illustrate some of the technical considerations in producing minimum care cotton fabrics.

Procedure

The 80 × 80 print cloth was singed, desized with enzyme, rinsed, and bleached with hydrogen peroxide in a beck. It was then mercerized at 2 in. under greige width with 26% sodium hydroxide at 30° C. followed by thorough rinsing with water at 85° C. and with 0.4% acetic acid. This preparation gave a fabric alkalinity of 0.15% calculated as sodium hydroxide. A portion of the fabric was then further neutralized in 2% acetic acid to give fabric alkalinity of 0.06% as sodium hydroxide. A portion of the mercerized fabric was washed with 0.3% sodium carbonate to give a higher fabric alkalinity of 0.3%.

The amount of additive material in the padding formulation will be called resin solids for lack of a better simple expression. The wet pickup was in all cases 65% of the resin-catalyst solution on the weight of the fabric. Padded samples were air-dried, then cured through a continuous curing oven at 140°, 160°, or 180° C. for 70 sec. The cured samples were washed in a rotary wash wheel with 0.2% of a nonionic detergent at 50° C. for 10 min., rinsed thoroughly, extracted by centrifuge, and ironed on a flat bed press. All samples were conditioned at 70° F. and 65% relative humidity for at least 48 hr. before testing by the TBL method. The average standard deviation of the mean of ten warp and ten filling measurements was approximately 0.03 cm. Breaking load was determined by Grab Method 5100 of Federal Specification CCC-T-191b on the model DH Scott Tensile Tester. The average standard deviation of the mean of ten samples was 0.7 lb.

Influence of Resin Type and Concentration

Resins used were the methylol derivative of cyclic ethylene urea from 2.25 moles of formaldehyde per mole of ethylene urea, and the methylol melamine from 2.5 moles of formaldehyde per mole of melamine.

Each point in Figure 3 represents the average of results obtained with two different catalysts, 2-amino-2-methyl-propanol-1 hydrochloride and magnesium chloride in equimolar concentrations. Under no condition of resin solids or curing temperature was a significant difference found at this level of fabric alkalinity for crease recovery or breaking load which would favor one catalyst over the other. The three connected points in Figure 3 indicate curing temperatures 140°, 160°, and 180° C. In each case, breaking load decreases with increased curing temperature.

Considering first the DMEU performance, note that the amount of resin determines the crease recovery level which is relatively independent of curing temperature in the range 140° to 180° C., but that as the temperature is increased there is a significant decrease in breaking load. This indicates the desirability of low temperature cure for this resin to get the best recovery–breaking load relationship.

The melamine formaldehyde results show no such clear-cut relationship between crease recovery and resin solids since curing temperature is here an imimportant variable. The improvement in crease recovery obtainable by a 40° increase in temperature is as great as the improvement obtained by a threefold increase in amount of resin. Previous investigators have pointed out that on a solids basis DMEU is more effective than MF in producing improved crease recovery, and these data confirm this. Six per cent of MF at the optimum temperature gives the same level as 4% DMEU (3.0 cm.). At the other extreme 18% MF gives the same level as 12% DMEU (3.5 cm.); or approximately 50% more MF is required to produce equivalent crease recovery. Actually the over-all results are far from equivalent since the breaking loads for melamine formaldehyde are significantly better than for DMEU, particularly at high levels of crease recovery which are of most interest in a discussion of minimum care properties. There is also an improved crease recovery–breaking load relationship for the highest amount of melamine formaldehyde solids. This improvement is not found as the amount of DMEU solids is increased.

Other investigators have suggested that for a given fabric there is a unique relationship between crease recovery and breaking load which is independent of type or amount of thermosetting resin. In other words, at any given level of crease recovery the strength is approximately equal for different resins. These data indicate a wide range of strength results obtainable at a given crease recovery, and this emphasizes the importance of the proper selection of chemicals and processing conditions to obtain optimum results.

Figure 4 shows the relationship between crease recovery and tear strength for the same experiments. These tests are by the Trapezoid Method 5136 of the Federal Specification, with the exception that the indicated strength is an over-all average instead of the average of the five highest peak loads. The

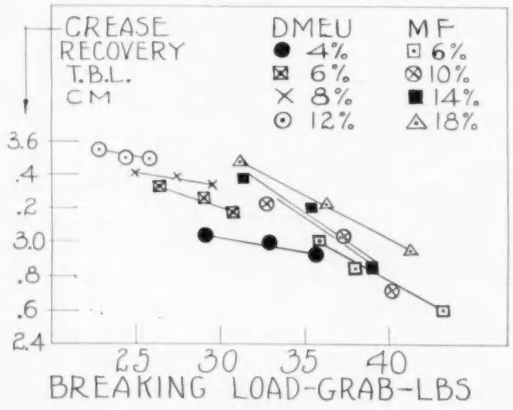

Fig. 3

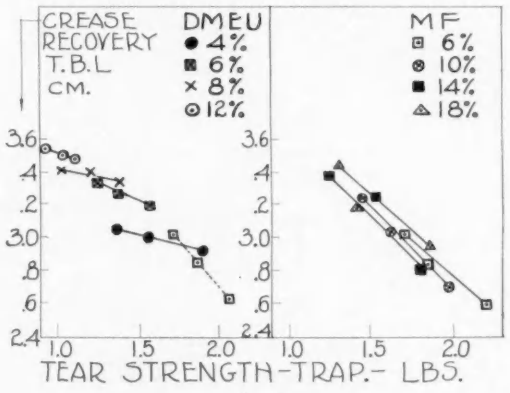

Fig. 4

data for the two resins are separated to avoid confusion, except that the 6% MF curve is inserted for reference as a dotted line on the DMEU data. The lines indicate increasing temperature toward the upper left. Again, note the distinct separation for DMEU depending on amount of solids, and also at any given solids note the rather substantial decrease in tear strength as the temperature is increased with almost no improvement in crease recovery. For MF there is no such separation based on resin solids since curing temperature is such an important determinant of crease recovery. Here again at high crease recovery, MF shows a 15 to 20% higher strength than DMEU.

Figure 5 is the same crease recovery–breaking load data plotted so as to emphasize the effect of temperature. The lines represent resin solids increasing toward the upper left. The lines are not intended to represent the shape of the curve but rather to aid the eye in following these data. The better performance of DMEU at low temperature cure than at high temperature of cure is again noted. Also with MF a high curing temperature is required to obtain levels of crease recovery above 3.3 cm.

Figure 6 is the crease recovery–tear strength data previously shown, plotted to emphasize the temperature influence. Again there is better recovery-strength relationship for the low curing temperature with DMEU. On the other hand, with MF, the crease recovery–tear strength relationship is practically constant for the range of temperatures and resin solids which has been investigated. Symbols used in Figures 5 and 6 correspond to those used in Figures 3 and 4.

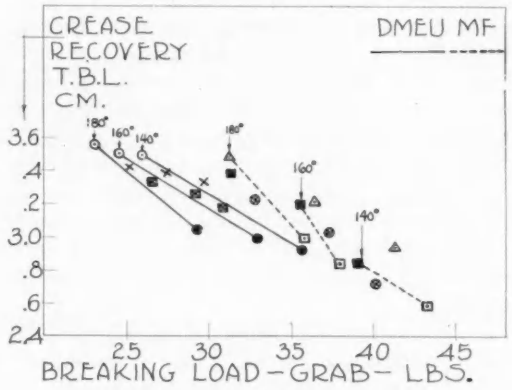

Fig. 5. Same symbols as in Fig. 3.

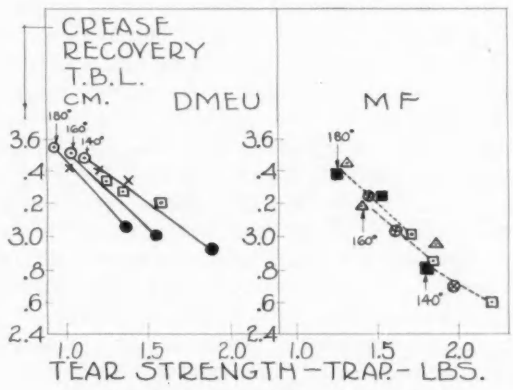

Fig. 6. Same symbols as in Fig. 4.

Influence of Catalyst Type and Concentration

Figure 7 indicates performance of 6% DMEU on fabric of low alkalinity, 0.06%. The three physical properties, crease recovery, breaking load, and tear strength are plotted separately as the ordinates against catalyst concentration in moles per liter. In all cases results at 160° are intermediate but are not shown for simplicity. Results are shown for three concentrations of catalyst, the middle one (0.036 moles per liter) was the catalyst concentration used in all the previous experiments with DMEU where resin solids were varied. For reference, this concentration (0.036 moles per liter) corresponds to 20% of the usual commercial concentration of this particular amine hydrochloride on the amount of resin used here. On this scale the lowest catalyst concentration is 10% on the weight of the resin and the highest is 40%. Under all these conditions, the two catalysts give equivalent results for crease recov-

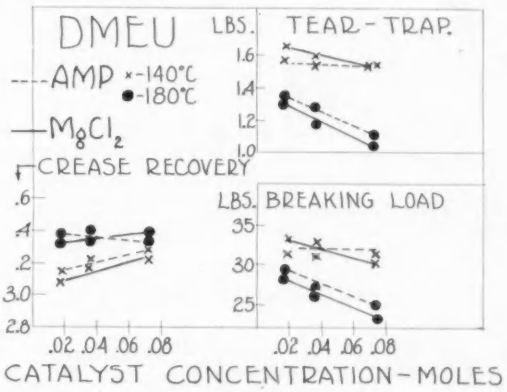

Fig. 7

ery, tear strength, and breaking load when used in equimolar concentration. This equivalence is fortuitous in view of the difference in mode of action of the two catalysts, even though both are of the so-called latent curing type. A detailed kinetic study would require additional data on the actual fiber temperature under ambient curing oven conditions and also data on the rate of moisture loss at temperatures in the effective curing range. The increase in catalyst concentration of either type has a moderate effect on crease recovery at the lowest temperature studied (140° C.) but no significant effect at high temperature. This is an indication that 140° C. represents a borderline curing condition which requires additional catalyst to obtain full effectiveness of the resin. The effect of increased catalyst concentration on strength is quite pronounced for the high curing temperature.

Figure 8 is from similar data for 10% melamine formaldehyde on the same fabric of low alkalinity (0.06%). Again the intermediate catalyst concentration, 0.06 moles per liter, represents the amount of catalyst used in the previous work where resin solids were varied. The three catalyst concentrations correspond to 10, 20, and 40% of the commercial amine hydrochloride solution of the weight of the resin in the padding solution. Here, in the presence of a much larger temperature effect on crease recovery than that found for DMEU, there is still a close equivalence of the two catalysts on an equimolar basis. There is no evidence of increased efficiency with magnesium chloride. Note that for melamine formaldehyde there is no evidence of any deleterious effect of high catalyst concentration on breaking load or tear strength such as was noted with DMEU.

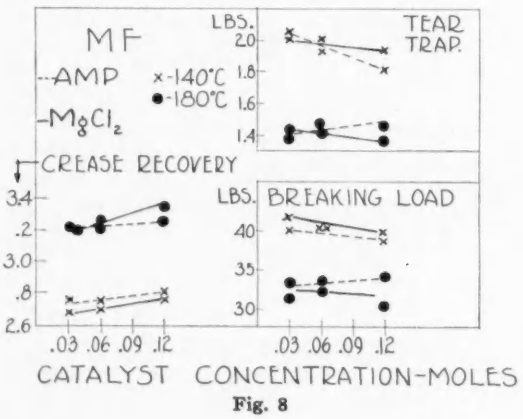

Fig. 8

Effect of Variation in Fabric Alkalinity

The range of fabric alkalinity for this work was selected to correspond to practical mill operating conditions. The 0.06% alkali concentration represents the range obtainable when neutralization is carried out on mercerized fabric as a separate operation in a high cloth capacity washer with a weak acid such as acetic. The 0.15% alkali concentration represents that expected when the neutralizing is done on a high speed mercerizing range with efficient cascades and suckers and low capacity wash boxes such as Williams units charged with acetic acid or sodium bicarbonate. The high alkalinity range, 0.3%, occurs when such a high speed mercerizing range is not operating efficiently, for example, with imperfect operation of cascades or suckers or when the alkali concentration of the wash boxes builds up.

The measurement of chlorine retention damage is by the Tentative AATCC Procedure. It should be mentioned that the committee responsible for the evaluation and improvement of this test method has recently published a report [1] of their findings that this test does not yield good interlaboratory agreement. However, the tentative test is the best now available for measurement of this important property of cotton finishes. In this laboratory, duplication within 10% breaking strength loss is consistently obtained.

From the data presented in the tables, it is evident that it is not proper to characterize a thermosetting resin in terms of its chlorine retention damage without specifying application conditions. Under the range of conditions, MF shows breaking strength losses ranging from 0 to 100%. The similar data for DMEU will show a range from 1 to 85%. The two most important variables influencing chlorine retention are temperature of cure and catalyst-fabric alkalinity balance. Table I emphasizes the effect of fabric alkalinity. At the lowest curing temperature the damage is uniformly high. At the higher curing temperature the damage is relatively low at 0.06 and 0.15% alkali but becomes high under some conditions at 0.3% alkali. The greatest difference between the two catalysts when used in equimolar quantities appears when the fabric alkalinity is high. At 0.3% alkali the chlorine retention loss with AMP is uniformly high and only at 180° C. is any crease recov-

TABLE I. Crease Recovery and Chlorine Retention Damage 10% Melamine Formaldehyde 0.06 Moles Per Liter of Catalyst

Fabric alkalinity % as NaOH	Curing temp. (° C.)	AMP·HCl C.R. (cm.)	AMP·HCl Cl damage (%)	$MgCl_2$ C.R. (cm.)	$MgCl_2$ Cl damage (%)
0.06	140	2.8	85	2.8	95
	160	3.1	30	3.0	25
	180	3.2	5	3.3	0
0.15	140	2.9	70	2.7	100
	160	3.1	25	3.1	35
	180	3.2	0	3.3	0
0.30	140	2.2	100	2.5	100
	160	2.3	100	2.9	70
	180	2.8	100	3.1	15

ery developed. The lower temperatures produce the crease recovery equivalent to untreated fabrics but with high chlorine retention damage. The magnesium chloride is more effective in maintaining crease recovery at high fabric alkalinity; at high curing temperature a low level of chlorine retention damage is maintained.

Table II indicates that increased catalyst concentration compensates for high fabric alkalinity. However, the appearance of high fabric alkalinity under mill operating conditions is often undetected. Only when a thorough acid sour is included in processing can high fabric alkalinity be completely avoided.

TABLE II. Crease Recovery and Chlorine Retention Damage 10% MF—Fabric Alkalinity: 0.3% as NaOH

Cat. conc. mole/liter	Curing temp. (° C.)	AMP·HCl C.R. (cm.)	AMP·HCl Cl damage (%)	$MgCl_2$ C.R. (cm.)	$MgCl_2$ Cl damage (%)
0.06	160	2.2	100	2.9	70
	180	2.8	100	3.1	15
0.12	160	3.1	35	3.2	15
	180	3.3	0	3.4	0

Table III indicates that the two catalysts have similar effect on DMEU under changing alkali concentration. Also with this resin, high fabric alkalinity can be compensated by increased catalyst, as shown in Table IV. Depending on conditions of finishing, DMEU exhibits chlorine retention strength loss of 1 to 85%.

In presenting all of these data, it has not been the intention to indicate any optimum finishing formulation to achieve minimum care properties. Instead the wide variation in results which can be realized has been pointed out. No mention of the secondary role of other chemical constituents in the padding formulation has been made, such as widely used thermoplastic resins which greatly modify the hand and drape, or the yarn and fiber lubricants which in some cases improve fabric tear strength. In all cases the finisher must select the optimum formulation based on his relative requirements for crease recovery, strength, durability, chlorine retention, and cost.

TABLE III. Crease Recovery and Chlorine Retention Damage 6% DMEU—0.036 Moles Per Liter of Catalyst

Fabric alkalinity % as NaOH	Curing temp. (° C.)	AMP·HCl C.R. (cm.)	AMP·HCl Cl damage (%)	$MgCl_2$ C.R. (cm.)	$MgCl_2$ Cl damage (%)
0.06	140	3.2	30	3.1	15
	160	3.2	10	3.3	15
	180	3.3	0	3.4	10
0.15	140	3.2	50	2.8	65
	160	3.3	5	3.1	35
	180	3.4	0	3.4	10
0.30	140	2.1	Resin	2.1	Resin
	160	2.1	washed out	2.2	washed out
	180	2.2		2.6	84%

TABLE IV. Crease Recovery and Chlorine Retention Damage 6% DMEU—Fabric Alkalinity: 0.3% as NaOH

Cat. conc. mole/liter	Curing temp. (° C.)	AMP·HCl C.R. (cm.)	AMP·HCl Cl damage	$MgCl_2$ C.R. (cm.)	$MgCl_2$ Cl damage
0.036	160	2.2	uncured	2.2	uncured
	180	2.3	uncured	2.6	84%
0.072	160	2.8	80%	3.1	50%
	180	3.2	35%	3.3	15%

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Manuscript received September 6, 1956.

Minimum Care Fabrics from Viscose Rayon[1]

T. R. Scott, Jr.[2]

I THINK that "minimum care fabric" represents a logical, realistic choice of terminology. Although the term "wash and wear" has obvious consumer appeal, we have already seen examples of very poorly finished garments being sold under the wash and wear label. Also, the question has been raised as to whether even the better finished garments on the market today are actually wash and wear for the most fastidious customers; perhaps all that this group would require is greater ease of care. Furthermore, those who are looking for no-iron garments are going to be dissatisfied if they have been misled by improper labeling.

Although relative freedom from wrinkles is a prime requisite, there are other equally important requirements. A minimum care garment should, after repeated washing, show good retention of dimensions, hand and color, and should have a reasonable service life.

It is well known that apparel fabrics knitted or woven from cellulosic yarns are outstanding in wearer comfort, particularly in garments which come in contact with the body. The physical characteristic of water absorbency which is largely responsible for highly satisfactory comfort for the wearer, however, presents some difficulties in rendering these fabrics crease resistant and dimensionally stable.

Rayon is an extremely hydrophilic fiber; that is, it readily imbibes large quantities of water. For example, when a typical rayon yarn is immersed in water, it will swell about 1% or more in length and from about 45 to 85% in diameter [5]. The longitudinal swelling is not much of a problem but the transverse swelling or increase in fiber diameter causes fabric shrinkage [6], and results in lower yarn strength when wet and lower resistance to wet abrasion. Thus, one of the problems involved is to find means of reducing fiber swelling, or water absorption. If water absorption is decreased, the fabric becomes more dimensionally stable [2], wet strength and abrasion resistance are increased, and the fabric will dry faster. All of these advantages will accrue while retaining the advantage of comfort for the wearer.

The requirement that minimum care fabrics must have a reasonably satisfactory appearance after drying, either on the line or in a home drying machine, brings us face-to-face with the question of how to measure this property in the laboratory. Apparently, the only way to test this property at present is the subjective test of washing and drying a fabric and then inspecting it for wrinkles. Various people are working on an objective test, and although certain proposals have been made, I am not aware that any such test is in use at present.

While there appears to be general agreement that the appearance of a garment after drying must be related in some manner to crease recovery, the most widely used methods of measuring crease recovery, viz., T.B.L. and Monsanto, cannot always be used as a basis for accurately predicting no-iron characteristics. It has been said that if a high crease recovery value is obtained on a stabilized fabric, one might suspect that this fabric has minimum care properties; a low value, on the other hand, does not preclude this possibility.

In considering resin treatments for dimensional stability and crease recovery, rayon appears to have a number of advantages.

1. Rayon frequently gains in wet tensile strength following minimum care treatment, while losses in dry tensile are usually slight. (In some cases there is an increase in dry tensile strength.)

2. The tear strengths of fabrics are invariably lowered by minimum care treatments. By use of a softener, either in the bath or in a subsequent operation, the tear strength of filament and staple rayon can be restored, provided the fabric has not suffered irreversible damage due to improper finishing techniques.

3. With respect to acid hydrolysis, which may occur during resin treatment or during chlorine bleaching followed by ironing of a treated fabric, rayon loses little or no strength under conditions where cotton not only loses strength but sometimes suffers irreversible acid damage [4].

[1] This paper is a condensation of the address by T. R. Scott, Jr., delivered at the T.R.I. Annual Meeting, March 22, 1956.

[2] Textile Operations Section, Textile Research Department, American Enka Corporation, Enka, N. C.

Types of Fabric Treatments Available

It is not the purpose of this paper to discuss in detail the chemistry and application of the many chemicals currently being used in various combinations to produce stabilized, crease-resistant minimum care fabrics. A list of the more commonly used compounds is, however, given in Table I.

TABLE I. Chemicals Used to Produce Stabilized, Crease-Resistant Cellulose Fabrics

1. Urea-formaldehyde (durability to washing questionable)
2. "Modified" urea-formaldehyde
3. Melamine formaldehyde or modified melamine resins
4. Formaldehyde plus cellulose ethers, starches, gums, or vinyl copolymers
5. Glyoxal plus urea-formaldehyde

Although all of the materials listed above can be made to give good dimensional stability and, in most cases, good crease recovery, some of them have serious shortcomings when their applicability in a minimum care formulation is considered. In addition to imparting a satisfactory appearance after drying, the finish should possess other important characteristics. It should (1) be durable to repeated laundering, (2) impart satisfactory dimensional stability and hand, (3) not seriously affect tensile and tear strengths or color, (4) not greatly reduce abrasion resistance, (5) not be chlorine-retentive to any appreciable extent.

The technical considerations in the application of minimum care finishes are the same as those involved in rendering rayon fabric dimensionally stable and crease-resistant. The proper control of these operations is vital to the production of high quality merchandise. We have seen examples of variations in the performance of fabrics finished with the same formulation in different mills as well as variations in the results found in different runs in the same mill; the former could possibly be attributed to minor differences in equipment while the latter must be caused by inadequate process control. Cost of treatment, including dyeing, might range from about 8 or 9 to 20 cents per square yard, depending upon the type of treatment employed, types of dyes used, and weight of the fabric. Figure 1 shows a yarn-dyed filament rayon garment which has received the rather drastic treatment of 20 washings at the boil.

This finish was of the acid-aldehyde type and is quite durable to repeated laundering, as shown by a shrinkage of less than 3% in warp and filling after 20 washings at the boil. Tensile and tear strengths were not appreciably affected, and resistance to abrasion—at least the type of abrasion encountered during washing—is increased. This can be seen by examining the collar and shoulder areas of the two garments; the control fabric is badly frayed while the treated garment shows only slight wear. This treatment also results in greater resistance to seam slippage; this is especially noticeable in these garments at the waist, where it can be seen that the treated garment is superior in this respect. Although this probably cannot be seen in the photograph, the treated garment shows excellent retention of color while the untreated garment is badly faded.

In addition to selection of the most suitable finish formulation, there are other factors to be considered. For example, fabric construction is fully as important as the finish used. The most satisfactory fabrics are the result of a combination of suitable fabric geometry with a good finish. It has been demonstrated that spun yarn fabrics which are of a loose, open weave, and which have a fair degree of crease resistance to begin with, are easiest to render crush-resistant [1]. Our experience has shown that such fabrics are also more amenable to the introduction of minimum care properties. Also, a wide variation has been found in the performance of various commercial treatments on different fabrics. The fact that a given finish is superior on one type fabric is no assurance that it will work best on another type. Thus, it might be said that, for any given application, most satisfactory performance will be realized by choosing the fabric and finish for the particular end use envisioned; experience acquired in one end-use area cannot always be successfully translated into another area.

Fig. 1. Treated and untreated filament rayon dresses after 20 washings at 212° F.

Filament versus Spun Rayon

We are familiar with fabrics made from spun rayon and from blends of rayon staple with cotton and various synthetics in shirting and suiting fabrics. As these fabrics have been adequately publicized, we will not discuss them in detail here [3 and 7]. Since, however, little if any data have been published on the use of filament rayon alone, or in combination with other fibers, I would like to discuss this further because this largely unexplored area appears to have good potentialities.

First, let us consider a 100% viscose rayon French crepe fabric, with 75-den. rayon in both warp and filling. Acid-aldehyde was used as the principal ingredient in the finish formulation.

It can be seen from Table II that this fabric has excellent dimensional stability, fair crease recovery, and satisfactory tear strength, both dry and wet. In addition, the treatment reduces the water retention to such an extent that the treated garment will dry in approximately one-half the time required for an untreated garment. This fabric, treated and untreated, was washed and tumble-dried. A photograph after drying, but with no ironing, is shown in Figure 2.

After inspection of the garment following washing and tumble-drying and after examining the physical properties of tensile and tear strengths, water retention, etc., we might conclude that this fabric meets the requirements for minimum care. When used in women's slips, possibly no ironing would be required. If this same material were used in women's blouses, however, they would probably require a light touching up with a hot iron. This is a good example of a case where a given fabric would be "no-iron" in one end use, whereas a light ironing would be required in another end use. In either use, however, the garment would dry faster, have good dimensional stability, and a pleasing hand, and these properties would be present even after repeated washing.

TABLE II. Physical Properties—Viscose Rayon French Crepe, 150 × 94

Warp: 75/60/3Z semidull filament rayon
Filling: 75/60/35Z filament rayon

Properties after 10 machine launderings at 160° F.	Untreated	Treated
Retention of dimensions, %		
Warp	96	99
Filling	81	99
Area	79	98
Monsanto crease recovery, %		
Warp	43	62
Filling	49	62
1-in. ravel strip strength, lb.		
Warp	25	24
Filling	27	22
Trapezoid tear strength, lb.		
Dry, warp	8.3	4.1
Dry, filling	7.3	3.7
Wet, warp	1.4	3.0
Wet, filling	2.4	4.5
Water retention, %		
Warp	103	34
Filling	90	36

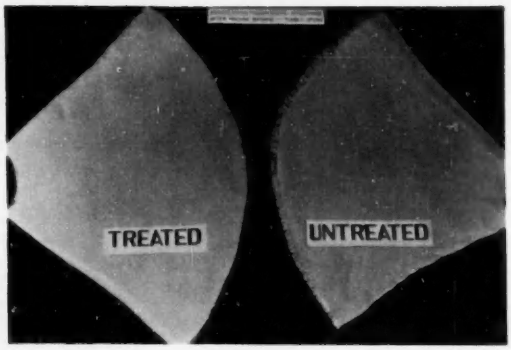

Fig. 2. Viscose rayon French crepe—appearance after machine washing and tumble drying.

A continuous filament fabric of this type with a smooth surface represents one of the most difficult types of material in which to introduce minimum care properties. This brings out another important point: Fabrics which have patterns or designs which tend to confuse the eye, such as printed, puckered, or plisséd surfaces, often present little difficulty in introducing minimum care properties because small wrinkles which may be present are not readily discernible to the eye.

We consider this 100% filament rayon fabric an interesting development and feel that it is an advance guard, so to speak, for minimum care fabrics of this type from filament rayon.

Blends with Other Fibers

In addition to the almost unlimited possibilities for blending viscose staple with other fibers, there are numerous applications for combination fabrics of spun and continuous filament rayon with other fibers. For example, we have a play clothing fabric woven from a cotton warp and a high tenacity 300-

den. filament rayon filling which has excellent minimum care characteristics. Some physical properties of this fabric are given in Table III. A modified urea-formaldehyde resin was used as the principal finish ingredient. In this table, it is seen that the treatment has produced good dimensional stability, a lower degree of water retention, and improved crease recovery. Figure 3 shows the treated fabric after machine washing and extracting followed by both tumble drying and line drying.

In finishing combination fabrics, the different behavior of various types of fibers toward resin-forming or cross-linking chemicals must be considered. Rayon can withstand quite satisfactorily certain finishing treatments which would seriously damage certain other fibers [4]; yet it is possible to select finishes which produce the desired results without undue damage to any of the component fibers.

The examples just cited indicate what might be accomplished in minimum care finishes for fabrics containing filament rayon. This application will require some effort, to be sure, because comparatively little has been done to date. We believe that the potential returns are great enough to justify this effort.

With regard to what the future holds for minimum care fabrics of viscose rayon, the work done to date gives us only a glimpse of the possibilities for using rayon in new applications. An example of such a new end use is a play clothing or denim fabric woven from solution-dyed continuous filament rayon in combination with cotton, although this fabric does not contain a minimum care finish. The result is truly striking.

TABLE III. Physical Properties of Combination Play Clothing Fabric

Warp: 18/1 carded cotton
Filling: 300/40 JETSPUN high-tenacity rayon

Properties after 20 machine launderings at 160° F.	Untreated	Treated
Retention of dimensions, %		
Warp	92.4	97.5
Filling	96.3	100.6
Area	89	98
Water retention, %		
Warp	39	30
Filling	64	44
Monsanto crease recovery, %		
Warp	52	68
Filling	52	61

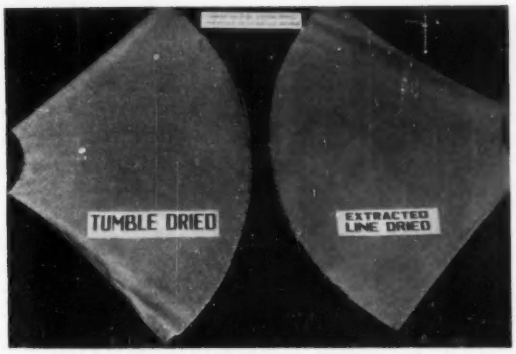

Fig. 3. Treated combination play clothing fabric after machine washing.

Now who would have thought of using rayon to lend glamor to such a prosaic fabric as denim? If this can be done with denims, shouldn't there be countless other applications where rayon could be used to supply plus values but isn't being used today simply because no one has ever thought of it?

The denim fabric with filament rayon in the filling has outstanding dimensional stability although this fabric was Sanforized only. Retention of dimensions after 20 washings at 160° F. were 99.9% in the warp and 99.4% in the filling. A similar fabric with cotton in the filling, which was washed under these same conditions, showed retention of dimensions of 100.5% in the warp and 97.9% in the filling.

Although most work to date on minimum care cellulosic fabrics has been done on spun rayons and cottons, substantial progress in this direction on filament rayon fabrics might be expected to open up such fields as women's house dresses, girls' everyday summer dresses, blouses, underwear, boys' shirts, men's work clothing and sport shirts.

Acknowledgments

The author wishes to thank L. Hart and L. R. Graybeal, of the American Enka Corporation, for supplying certain data used in the preparation of this paper.

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Industrial Textiles[1]

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INDUSTRIAL textiles make up a large segment of all textiles made in the United States, approximately 30%, of which over two-thirds is used in three areas, the rubber industry, bags and bagging, cordage and twine. Of the variety of raw materials employed in recent years, jute, cotton, and man-made fibers have shared in the proportion of one-third each. However, these may change if a recent prediction proves true, that 70% of the cotton duck used in the industrial field will be replaced within five years with coated man-made fiber fabrics.

Constructions range from dust cloths, weighing 1 oz./sq. yd., to belt ducks, weighing 41 oz./sq. yd. Webbings go as high as 72 oz./sq. yd. Tensile strength may be as low as a few pounds per inch of width, or as high as 3000 lb.

As to properties for industrial textiles, strength, controlled dimensions, flexing, and adhesion are among the most important. Textiles made from staple fibers usually provide better adhesion than filament materials because of fiber ends. The use of adhesives is often necessary. Open weaves permit the coating materials to strike through. For these properties the manufacturers usually work to rigid specifications requiring strict quality control and well-equipped testing laboratories.

As might be expected, growth of or increase in production has perhaps been slower than for such textiles as household or apparel fabrics, since they are not affected by fashions, are dependent on the growth of other industries, and substantial investment is involved in special equipment such as heavy looms or machines of special design. Figure 1, for example, shows a machine for wrapping braiding of heavy-ply yarns around rubber hose to give it strength.

Of the many textiles used in rubber products, the manufacture of tire cord in particular has employed a succession of raw materials to the steady improve-

Fig. 1. Machine for wrapping braiding of heavy ply yarns around rubber hose to give it strength.

[1] Presented at the 26th Annual Meeting of Textile Research Institute, March 23, 1956.
[2] Development Manager, Textile Division.

ment of tire quality and reduction of its cost. Tire cost per mile is now less than 5% of the cost in 1906.

At that time an automobile owner got something like 53 miles per tire dollar. By 1927, this figure was raised to 432 miles per tire dollar. In 1937, it climbed to 800 miles. Today, the motorist receives over 1,600 miles for every dollar he spends for a tire.

Robert William Thomson, an Englishman, built the first pneumatic tire in 1845. He used leather to cover and protect the rubber air tube and to give the tire strength. Thomson's tire met with little success.

In 1888, John Boyd Dunlop, of Belfast, Ireland, built his first tire. It consisted of a rubber air tube with a linen jacket. The outside was covered with rubber sheeting.

The first reinforcing material for pneumatic tires was square woven cotton duck, usually 17¼ oz./sq. yd., and woven with 23 ends and 23 picks of 23/11 ply, made of combed Sea Island or Egyptian cotton.

The first radical changes in reinforcement came about in 1914 with the development of cotton tire cord, which, instead of having equal strength in both directions, had strength in the warp direction only, the fine filling yarns functioning only as a means of spacing and holding the warp yarns together. Almost immediately the carcass began to last as long as the tread. Figure 2 shows the special creel arrangement for the first twisting operation in making tire cord. The yarn is wound on a beam instead of the conventional spool or tube.

However, in spite of the higher level of tire mileage afforded by cord fabrics, tires continued to fail

Fig. 2. Special creel arrangement for first twisting operation in making tire cord.

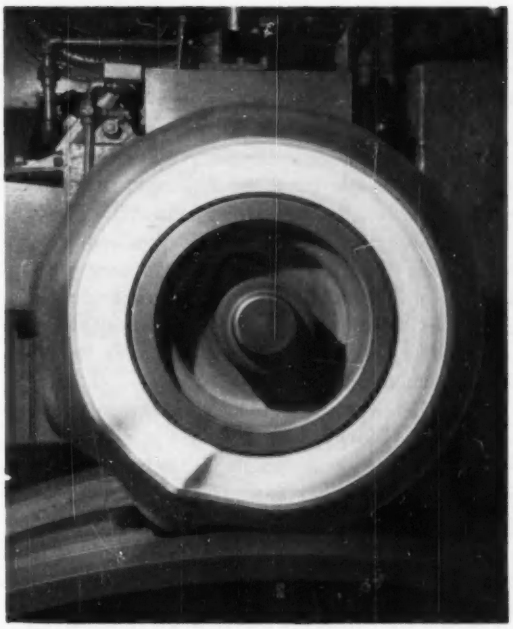

Fig. 3. Distortion of tire striking a test wheel at a speed of 200 m.p.h.

as the result of flex fatigue. It has been calculated that every cord in a tire bends and twists more than half a million times every 1000 miles. The tire illustrated in Figure 3, striking a test wheel at 200 m.p.h., shows distortion nearly halfway around its circumference.

In the early 20's higher twists in both ply and cable were added to the tire cord specification to improve flex life. Previous constructions for maximum strength were more or less standard—23/5/3, having 17½ t.p.i. in the ply and 7½ t.p.i. in the cable. Ply and cable twists were increased to 21 and 9½ t.p.i. respectively, which provided a compromise between strength and maximum fatigue resistance.

Not only was quality improved, but costs were reduced, because now shorter staple cotton could be used. $1\frac{1}{32}$ to $1\frac{1}{16}$ American cottons replaced the more costly Sea Island and Egyptian cottons. Combing was discontinued and carded yarns came into general use. The construction was modified by replacing 23/5/3 cords with lower cost 12/4/2 ply.

One serious problem remained. Higher twist gave the cords high elongation, which permitted the tire to "grow" in cross section. Pulling out some of this stretch by tensioning the cord before it was put

into the tire was found to be the answer. Elongation at the 10-lb. load was reduced from 12% to 6 to 8%.

High-tenacity viscose rayon was first introduced in the middle 30's. Then came World War II. Cotton was called upon for many military jobs in addition to tires. A better fiber was needed to reinforce tires made from the new synthetic rubber. High strength rayon provided the answer. By 1943, rayon was generally adopted for automobile and truck tires. Like cotton cord, it too went through a series of improvements. The finer deniers with high plies were replaced with heavier deniers and fewer plies. Today, 1650 denier 2 ply is the standard construction.

In 1953, new super high-strength rayons went into production. Now nylon, first used in airplane tires, is making a major impact on the automotive industry because of its strength and outstanding bruise and impact resistance.

Tires are now being made with a layer of steel wire cord fabric between the rubber tread and the four plies of nylon cord to make the thread invulnerable to cut, rupture, or blowout. Figure 4 shows the tread cut away from a tire, exposing the two criss-cross layers of wire cord beneath.

Fig. 4. High strength steel wire is used in a new line of safety passenger and truck tires developed by U.S. Rubber Co. Section of tread cut away exposing two criss-cross layers of wire cord.

Tubeless tires brought new problems in tire design. Cotton chafer fabric is giving way to thinner, stronger, special chafers made from high-tenacity rayon and nylon. This fabric is called chafer because it is used around the bead of the tire to take any friction between the bead and the rim of the wheel.

Another industrial textile of interest is Ustex, a chemically and mechanically treated cotton yarn developed in the laboratories of the United States Rubber Company. Started in the late 1920's, it was at first a chemical treatment giving strength improvement of 8 to 10%. Later, tensioning increased this strength advantage in tire cord to 35 to 40%. It was not used in tires, however, because its low elongation had no "give." But softer twisted ply yarns increased in strength 75 to 85%. Table I compares the properties of untreated and Ustex-treated yarns.

TABLE I

	Before treatment		After treatment	
		Rtg.		Rtg.
Construction	9.5/9 ply		10.5/9 ply	
Tensile strength, lb.	23.8	100	38.4	161.3
Yd./lb.	872	100	950	108.9
G./den.	2.10	100	3.70	176.2
Elongation at 10 lb., %	4.79	100	1.25	26.0
Diameter, in.	0.0362	100	0.0305	84.3
Twist, t.p.i.	3.09	100	2.53	81.9

Ustex yarns are always plied—in Table I, 9 plies. Tensile is increased 61%, yards per pound 9%, with an increase in g./den. of 76%. These higher strengths can be translated into cost savings based on price per 1000 yd. used. Table II shows equal strength at 18% lower cost.

TABLE II

	Conventional		Ustex	
		Rtg.		Rtg.
Construction	8/10 ply		10/7 ply	
Tensile req.	27.0	100	27.0	100
Elongation at 10 lb., %	7.9		1.5	
Gauge	0.0335	100	0.025	75
Yards/lb.	672	100	1200	178
Price/lb., ¢	84	100	1.23	147
Cost/1000 yd.	1.25	100	1.025	82

In conveyor belts, the trend is toward greater length and steeper installations. Figure 5 shows a

TABLE III

	48-oz. Cotton duck		Ustex—nylon	
		Rtg.		Rtg.
Construction				
Warp	6/11 ply		8/8-ply Ustex	
Filling	6.75/6 ply		840 den. 3-ply nylon	
Count				
Warp E.P.I.	22	100	32	145.2
Filling P.P.I.	12	100	8.5	70.8
Gauge, in.	0.086	100	0.061	70.9
Wt., oz./sq. yd.	41.0	100	27.5 (33% lighter)	67.1
Tensile grab				
Warp, lb.	910	100	1250 (37% stronger)	137.4
Filling, lb.	275	100	308	112
Strength, lb./oz. wt.	28.9	100	56.7	196.2
Belt strength/in./ply	480	100	938	195
Strength/oz. wt./in./ply	11.7	100	34.1	291.5
Fabric cost		100		62.3

Fig. 5. Typical installation of long installation of conveyor belt.

typical installation. It has been stated that installations 100 miles long are not far from reality.

Although 32-oz. cotton duck was heavy enough in 1920, the next step was 36-, 42-, and finally 48-oz. ducks. Belts made from heavier ducks did not trough properly, and yet belt manufacturers wanted to increase belt ratings from 60 to 150 lb./in. of width per ply—an increase of 150%. In Table III the properties of a 48-oz. cotton duck of 6/11 ply warp, 6.75/6 ply filling are compared with a belt of Ustex warp 8/8 ply and 3-ply nylon filling.

Excellent results have also been obtained when Ustex yarns are used in braided high-pressure hose. Here it is not uncommon for two plies of Ustex to exceed the bursting strength of three plies of conventional yarn, and at reduced cost to the manufacturer.

In this very brief discussion an attempt has been made to give some idea of the importance of industrial textiles in our economy and the progress through research in the development of improved products at lower cost.

Manuscript received April 18, 1956.

The Home Laundering of Wash and Wear Garments of Hydrophobic Fibers[1]

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Introduction

It has been known that properly prepared garments made from certain of the hydrophobic fibers possess the following "ease-of-care" properties.

1. Retention of neat appearance during wear
2. Ability to be worn, washed, and worn again with only an occasional pressing
3. Retention of pleats and creases during washing and drying
4. Rapid drying
5. Good dimensional stability
6. High degree of durability
7. Resistance to degradation by moths and mildew

In an attempt to define the conditions necessary to obtain the maximum ease-of-care performance, a systematic study has been carried out on the effect of fabric construction and laundering variables on the ease-of-care properties afforded by the three hydrophobic fibers, "Dacron"[2] polyester fiber, "Orlon"[2] acrylic fiber, and nylon.

The laundry study was concerned mainly with the use of the automatic washing machine and dryer because it was believed that the optimum in ease-of-care will come from the proper combination of well-constructed and finished fabrics with completely mechanized home laundering.

Description of Fabrics Used in Laundry Study

The fabrics used for this study included spun yarn and filament yarn fabrics of 100% Dacron polyester

[1] The information covered in this paper was presented before the Annual Meeting of the Textile Research Institute, March 1956, by R. W. Peterson, Research Director, Textile and Industrial Products Research Division, E. I. du Pont de Nemours & Co., Inc.

[2] Du Pont trademark.

TABLE I. Representative Fabrics of Hydrophobic Fibers Evaluated in Laundry Studies

100% Dacron	Filament shirting—Oxford
100% Dacron	Spun shirting—batiste
100% Orlon	Spun shirting—broadcloth
70/30 blend of Dacron and cotton	Spun shirting—broadcloth
70/30 blend of Dacron and rayon	Spun shirting—broadcloth
65/35 blend of Dacron and cotton	Spun shirting—Oxford
90/10 blend of nylon and rayon	Spun shirting—broadcloth
100% Dacron	Filament shirting—plain weave
100% nylon	Filament shirting—plain weave
100% Dacron-Taslan*-textured	Filament shirting—Oxford
100% Dacron	Spun suiting—tropical
100% Orlon	Spun suiting—tropical
85/15 blend of Dacron and nylon	Spun and filament suiting—cord
75/25 blend of Dacron and cotton	Spun and filament suiting—cord
75/25 blend of Dacron and Orlon	Filament suiting—cord
50/50 blend of Dacron and cotton	Spun and filament suiting—poplin
75/25 blend of Orlon and nylon	Filament suiting—cord
60/40 blend of Orlon and cotton	Spun and filament suiting—cord
80/20 blend of Orlon and wool	Spun dress fabric—jersey
55/45 blend of Dacron and rayon	Spun suiting—linen weave
100% Orlon	Spun and filament suiting—tropical
75/25 blend of Orlon and cotton	Spun suiting—cord
65/35 blend of Dacron and rayon	Spun suiting—gabardine
70/30 blend of Orlon and wool	Spun suiting—flannel
100% Dacron	Filament dress fabric—faille

* Trademark for yarn textured according to quality standards set by du Pont.

fiber, Orlon acrylic fiber, or nylon; blends of these fibers with rayon, cotton, or wool; fabrics of the natural fibers; and resin-treated fabrics. A list of representative test fabrics made of hydrophobic fibers used in this work is given in Table I. Since printed patterns camouflage wrinkles, white fabrics or fabrics dyed a solid color were used in this study wherever possible. Although a number of complete garments were evaluated, most of the testing was done with 2×3-ft. fabric swatches.

Evaluation Techniques

A photographic technique was used in evaluating the laundering procedures to maintain a record and to facilitate comparison of results. A photograph of a fabric swatch, illuminated at an angle of 20 deg. to emphasize wrinkling, was taken after each laundering condition. These photographs were rated by a panel of five evaluators using a photographic scale of 1 to 5 (Figure 1). This scale was established so that 1 represented the photographed appearance of an ironed fabric, 2 represented the photographed appearance of slightly wrinkled fabrics still rated as acceptable for wear, 3 represented fabrics of borderline wearability, 4 represented fabrics which would definitely require some ironing to be acceptable, and 5 represented a very wrinkled fabric.

Effect of Washing Temperature on Wrinkling

The first variable studied was the temperature of the wash water. A full cycle of an agitator-type washing machine was used, including centrifugal extraction at 550 r.p.m. The fabrics were then line-dried at room temperature. Results from such washings at several different temperatures are shown in Figure 2. The data represent the average result for the large number of different wash-wear fabrics used. Although there were differences in the performance of these fabrics relative to one another, all fabrics responded in the same direction to changes in washing temperature.

When 140° F. water was used, all fabrics were severely wrinkled. Lower wash water temperatures resulted in a lower degree of wrinkling. When cold tap water at a temperature of 60° F. was used, most of the fabrics of hydrophobic fibers were acceptable for wear without ironing.

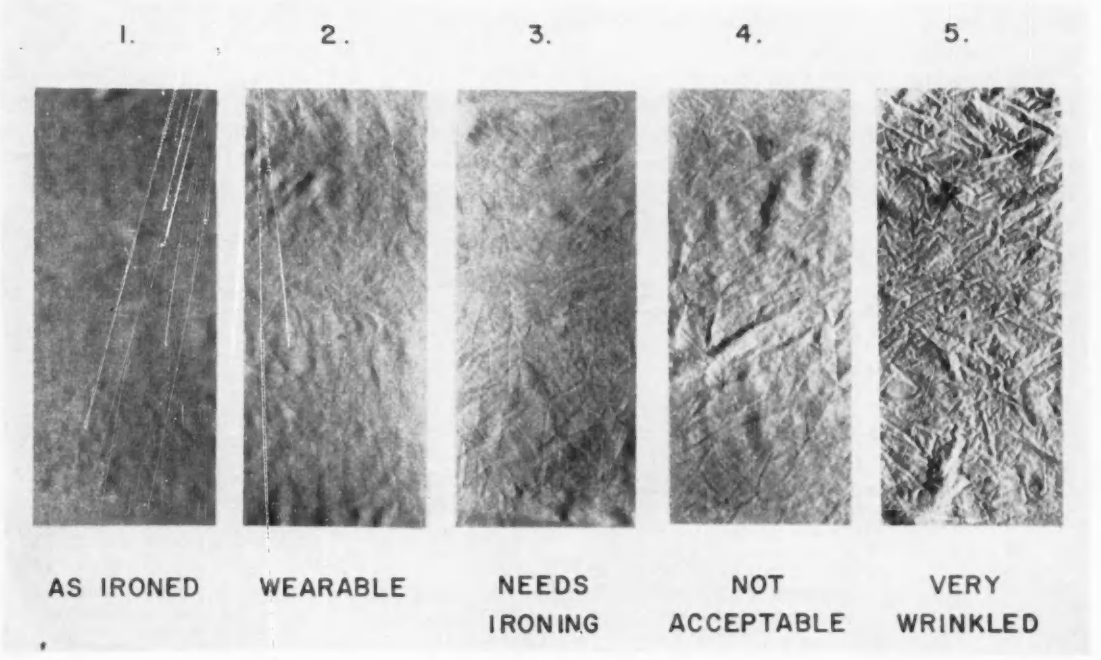


Fig. 1. Wrinkling scale.

In view of the success of the cold water washing in avoiding wrinkling, a study was made of the effectiveness of various detergents in cold water. It was found that several "built" household soaps gave as good detergency in 60° F. water as some popular synthetic detergents gave in 140° F. water.

Thus, it is not wholly unlikely that a satisfactory system of washing with cold water might be worked out as a means of reducing the cost of washing and of preventing wrinkling during washing. In this direction, indication was obtained that certain alkaline builders, such as sodium meta-silicate and sodium carbonate, can enhance detergency of soaps in cold water, apparently by substituting chemical action for thermal action.

It should be emphasized, however, that of all the detergent systems tested, better detergency was obtained with a given system at 100° F. than at 60° F. Furthermore, in field tests it was observed that when dress shirts of Dacron polyester fiber or Orlon acrylic fiber were washed repeatedly at 60° F., wrinkles gradually built up in that part of the shirt which had been tucked under the trouser belt. This did not occur with shirts washed at 100° to 120° F. Apparently mechanical agitation of the fabric in the warmer water removes these wear wrinkles. In the case of nylon shirts, however, wear wrinkles were removed even by cold water washing.

The important wrinkle-producing action in washing is the compression of the fabrics during draining and centrifuging while they are hot. When the fabrics are removed from the washing machine before the spin cycle and allowed to "drip dry," they are essentially free of wrinkles. A very successful washing technique for removing wear wrinkles and avoiding other wrinkles is to wash the fabrics in warm water, 100° to 120° F., then to replace the warm water with cold water (60° F.) by overflow rinsing while continuing the agitation, and finally to complete the automatic cycle using cold water for the rinses. This technique results in fabrics which rate as well (1.8 shown in Figure 2) as those which are agitated and spin-dried in 60° F. water.

Effect of Tumble Drying at Various Temperatures on Wrinkle Recovery

It has been found that the tumble dryer is one of the most effective means for promoting ease-of-care in garments of hydrophobic fibers. The same fabrics which had been tested in the washing study were subjected to further laundry experiments in which they were washed through the full cycle of an automatic washing machine at 100° F. These conditions, of course, led to considerable wrinkling of the fabrics as they were removed from the washer. The fabrics were then tumble-dried in a household dryer at several different temperatures. As the operating temperature was increased, the appearance of the tumble-dried fabrics improved. Wrinkles were effectively removed from fabrics of nylon at 130° to 180° F., from fabrics of Orlon at 140° to 180° F., and from fabrics of Dacron at 150° to 180° F. Tumble drying at appreciably higher temperatures (190° to 210° F.) introduced many wrinkles (see Figure 3). Since no serious wrinkling is introduced with any of these fibers until the temperature exceeds 180° F., it appears that the optimum temperature range for wrinkle removal by tumble drying is 170° ± 10° F. The operating temperatures discussed above were measured in the exhaust duct of

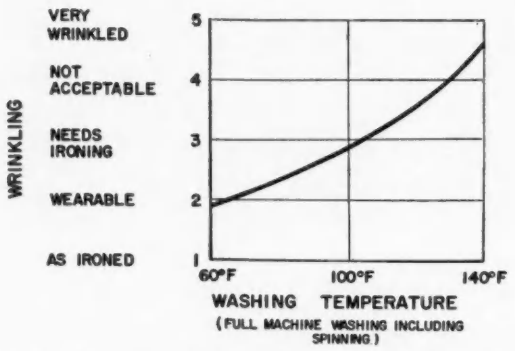


Fig. 2. Effect of water temperature during laundering on wrinkling. (Average of 25 fabrics.)

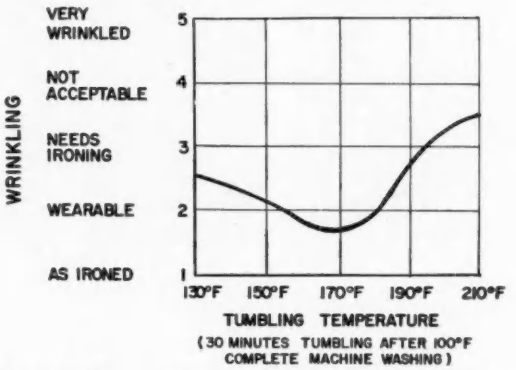


Fig. 3. Effect of temperature during tumble drying on wrinkling. (Average of 25 fabrics.)

the dryer. After drying, the actual temperatures reached by the fabrics in the dryer were 20° to 30° higher than the exhaust air temperatures noted.

Although four makes of dryers have been tested and found to be most effective at the 170° F. operating temperature, it is possible that other dryers, because of mechanical differences, may require slightly different exhaust temperatures for optimum effectiveness.

To avoid the wrinkling which occurs if the fabrics are allowed to stand under pressure while hot, the blower and tumbling action must continue for 5 to 10 min. after the heating current is shut off. This permits the clothes to be cooled while they are still being tumbled.

Good mechanical action was found necessary for wrinkle removal. Garments tumble more vigorously than sample fabric swatches, thus wrinkle removal was more effective on the former. A full load (9 lb.), consisting of 18 shirts, worked well. In the case of other types of garments where entangling was encountered, a lighter load was necessary to ensure adequate tumbling.

With the dryer used in these tests, a 30- to 35-min. drying cycle was found to be adequate for loads of 5 lb. or less of the fabrics studied. This provided for about 10 min. of actual drying time, about 15 min. of tumbling at the optimum temperature of 170° F., and about 5 to 10 min. of air cooling.

The tumble dryer worked equally well in removing wrinkles from garments that had been washed using hot (140° F.) water through the full cycle of the automatic washing machine, from garments that had been washed in cold water but which exhibited wear wrinkles around the belt line, and from wrinkled dry garments that had previously been line-dried.

It should be noted that creases or pleats which had been properly ironed into these fabrics persisted throughout repeated washing and tumble drying cycles. This is illustrated in Figure 4 which shows a fabric of Dacron and nylon with one crease that has been ironed in and one crease that has been formed by folding the fabric and placing a 160-lb. weight on the fold for two days to simulate a severe wear wrinkle. The third panel in Figure 4 shows the same fabric after it has been tumbled dry at 170° F. for ½ hr. The desired ironed-in crease is still sharp, while the simulated wear wrinkle has completely disappeared.

The center panel shows an identical fabric after being washed in an automatic washing machine, using warm water, and the complete cycle including spinning. As shown, the wash water removed the simulated wear wrinkle, but the fabric was generally wrinkled from spinning while it was warm, and the pressed-in crease was partially removed. When this wrinkled fabric was tumble-dried, it appeared identical to the third sample, i.e., completely free of

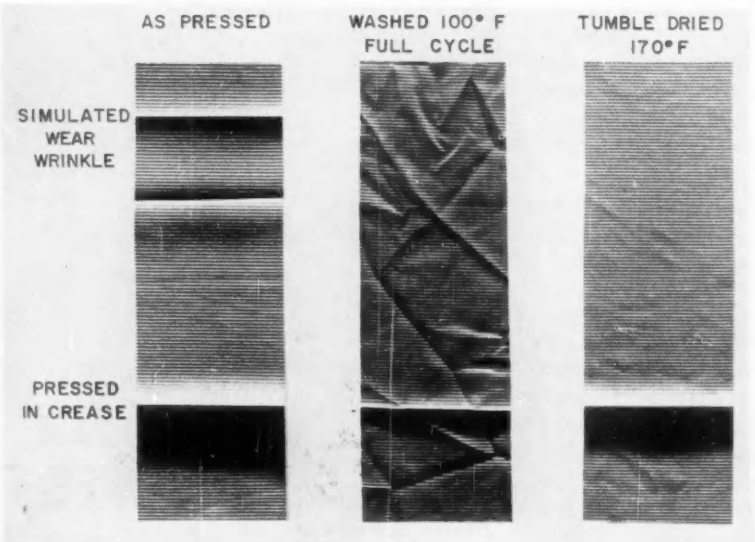


Fig. 4. Laundering of cord suiting of 85/15 Dacron/nylon.

wrinkles and with the desired pressed-in crease remaining.

It is believed that the action of these fibers in recovering original pressed form—whether it be flat or creased—on being heated and tumbled can be explained on the basis of "plastic memory." Fabric deformations occurring at temperatures below the pressing or setting temperature result in only semipermanent displacements of the molecules in the fiber. There is a tendency for these molecules to recover to their equilibrium positions even at room temperature, but the recovery rate is very slow. Heating the fiber to a temperature in the range of its second-order transition temperature hastens this recovery. However, we have shown in other experiments that simply heating a fabric is not sufficient to effect complete recovery of a wrinkle. It is postulated that this is the result of friction between fibers and yarns. Mechanical agitation while the fabric is hot acts to overcome the effect of this interfiber friction and allows the fibers to return to their former equilibrium positions. These actions, combined with the fact that moisture is not required, makes it possible for the tumble dryer to remove wrinkles from fabrics of Dacron polyester fiber, Orlon acrylic fiber, and nylon. Nine makes of dryers now on the market can be operated under the proper conditions to give this automatic ironing action.

Effort Required to Iron Various Fabrics

Many garments require ironing regardless of washing conditions used, and even the best of wash-wear garments require ironing when washed under adverse conditions. Tests have been conducted, using the Lauru platform [1] (Figure 5), to measure the amount of effort required to iron various commercial shirts. The Lauru platform consists of a plate mounted on piezoelectric crystals which generate a potential in proportion to the force exerted by a person standing on the platform. In the experiments reported below, the operator, while ironing, stood on the Lauru platform and used an ironing board which straddled the platform and rested on the floor. The results of a series of tests on business shirts are shown in Table II. Four shirts were evaluated: a batiste of 100% spun Dacron, a batiste of 65/35 blend of Dacron/cotton, an oxford of 100% Dacron filament yarn, and a prestarched cotton broadcloth. The shirts were washed in an automatic washing machine at 100° F., using the complete cycle, and were line-dried. The shirts, wrinkled as a result of washing, were ironed under the conditions believed optimum for each, i.e., a steam iron for the shirts containing Dacron and a dry iron for the dampened cotton shirt.

Shown in the last column of Table II are typical tracings from the recorder of the Lauru platform. The area under the force versus time curve is proportional to the physiological effort expended while ironing.

The second column in Table II shows the relative amount of work recorded for the vertical forces when ironing the several shirts. The batiste of Dacron required only 1/48 as much work as the cotton shirts, while the other two shirts containing Dacron required about 1/6 as much work as the cotton shirt. It required approximately 3 min. to iron the shirts of Dacron and approximately 7 min. for the cotton shirt.

Effect of Fiber Content and Fabric Construction on Ease-Of-Care Properties

The effect of several construction and finishing variables on the ease-of-care properties of fabrics from the hydrophobic fibers has been studied to a limited extent. Although this work is as yet incomplete, several tentative conclusions have been drawn.

1. In promoting ease-of-care performance, Dacron is the best of the fibers tested to date. This performance appears to stem from its high crease recovery,

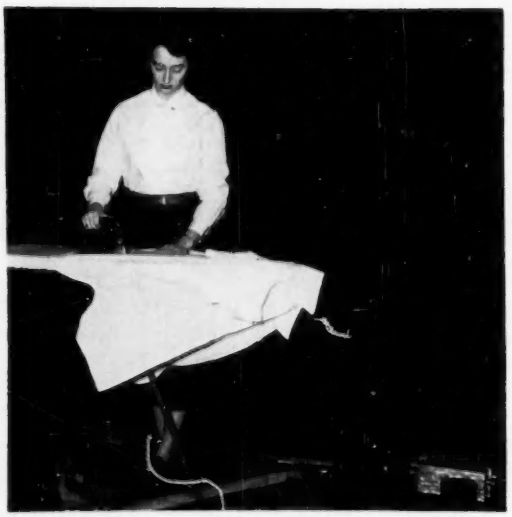
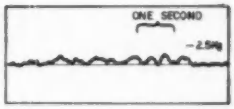
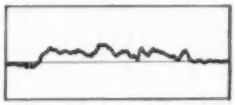
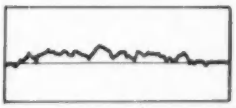
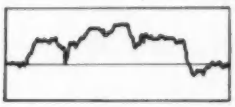


Fig. 5. The Lauru platform.

TABLE II. Effort of Ironing, Lauru Platform

SHIRT TYPE	TOTAL IRONING TIME (MINUTES)	TOTAL EFFORT (KG FORCE X SECONDS)	TYPICAL TRACING
100% "Dacron"– Batiste	3.2	23	
65/35 "Dacron"/ Cotton-Batiste	3.1	166	
100% "Dacron"- Filament-Oxford	3.2	178	
Cotton – Broadcloth	7.2	1115	

its relative insensitivity to water, its low rate of relaxation under stress, and its marked increase in rate of recovery when heated and mechanically agitated.

2. Increasing the filament denier improves the wash-wear performance of shirting-weight fabrics of either spun or filament yarns. This is probably due to the increase in the stiffness-to-friction ratio of the yarn, resulting in both better resistance to deformation and less hindrance to recovery.

3. Neither an increase in twist from 6 to 24 t.p.i. nor the plying of yarns showed any significant effect on the wash-wear performance of fabrics of Dacron filament yarn.

4. Fabric construction is an important variable. Studies of a series of fabrics have shown that weaves which permit more movement of the yarns increase wash-wear performance considerably. A marked improvement is obtained, for example, in going from a plain to an Oxford weave; a 2×2 basket weave is even better, and a 3×3 basket is the best. This effect is illustrated by the observation that a 2×2 basket weave of a blend of 65% Dacron and 35% cotton showed better wash-wear performance than broadcloths or batistes of 100% Dacron in spite of the better basic properties of the 100% Dacron yarn. Tricot fabrics of nylon or Dacron show excellent performance, presumably because of the relative looseness of the tricot construction.

Summary and Conclusions

The ultimate in ease-of-care properties is wash-no-iron performance. This can be obtained with properly made garments using hydrophobic fibers laundered at home in automatic washers and dryers. Where no tumble dryer is available, the optimum machine wash cycle would be one where the garments are agitated in warm water (100° to 120° F.), the water temperature reduced to 60° F by adding cold water while continuing agitation, and the complete cycle continued as usual, including spinning, using cold water for all rinses. Where a tumble dryer is available, the optimum laundry procedure would be to wash by any method at temperatures up to 120° F. and then to tumble dry at 170° ± 10° F. for 25 min., followed by 5- to 10-min. tumbling with cool air.

Tumbling in a dryer at this temperature was also found to be effective in removing wrinkles from dry garments of the hydrophobic fibers.

Measurements of the effort required to iron dress shirts showed that garments containing 100% or 65% Dacron polyester fiber were much easier to iron than conventional cotton shirts.

Ease-of-care performance of fabrics of hydrophobic fibers can be obtained by the proper choice of fiber composition and yarn and fabric construction. Fabrics of Dacron, Orlon, and nylon have shown wash-no-iron performance.

Acknowledgments

We wish to acknowledge the guidance of R. M. Hoffman and A. G. Scroggie in planning these experiments and the assistance of Mrs. B. J. McElroy and Mrs. I. M. Pugh in their execution. In addition, we wish to acknowledge the cooperation of L. A. Brouha and his co-workers in the experiments on the Lauru platform.

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1. Brouha, L. A., The Lauru Platform, in "Physiological Techniques in Work Measurements." *Proceedings of Annual Meeting,* Society for the Advancement of Management, New York, April 1956.

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Book Reviews

Modern Mill Controls in the Management of Production, Quality, Waste and Maintenance. Norbert Lloyd Enrick, New York, Modern Textiles Magazine, 1956. 28 pages. Price $2.00.

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It is believed that the answers found for cotton as a result of these studies will serve equally well for inspection and grading of many other products.

THE INDEX

TO THIS VOLUME HAS BEEN REMOVED FROM THIS POSITION AND PLACED AT THE BEGINNING OF THE FILM FOR THE CONVENIENCE OF READERS.

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